EAST Search History

Ref #	Hits	Search Query	DBs	Default Operator	Plurals	Time Stamp
L1	2	("5183911").PN.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	OFF	2008/01/16 18:19
S1	5	("3504038").PN.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	OFF	2007/09/22 15:46
S2	4	("2865937").PN.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	OFF	2007/09/22 14:38
S3	1	((JESUS) near2 (MIRABAL)).INV.	US-PGPUB; USPAT; USOCR	OR	ON	2008/01/16 15:41
S4	1	(("SILVIA AMPARO") near2 (CEPERO)).INV.	US-PGPUB; USPAT; USOCR	OR	ON	2008/01/16 15:41
S5	1	((VILFREDO) near2 (RUBI)).INV.	US-PGPUB; USPAT; USOCR	OR	ON	2008/01/16 15:42
S6	1	(("LIDIA ASELA") near2 (GARCIA)).INV.	US-PGPUB; USPAT; USOCR	OR	ON	2008/01/16 15:42
S7	1	((OSCAR) near2 (LOZANO)).INV.	US-PGPUB; USPAT; USOCR	OR	ON	2008/01/16 15:42
S8	3	((MARITZA) near2 (GOMEZ)).INV.	US-PGPUB; USPAT; USOCR	OR	ON	2008/01/16 15:43
S9	1	(("IRENE DE LA") near2 (LASTRE)).INV.	US-PGPUB; USPAT; USOCR	OR	ON	2008/01/16 15:44

EAST Search History

S10	2812	(fat? oil?) same (ozone or ozonation or ozonolysis)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2008/01/16 15:46
S11	2163	S10 and water	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2008/01/16 15:46
S12	916	S10 and peroxide	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2008/01/16 15:47
S13	286	S12 and emulsion	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2008/01/16 15:47
S14	58797	bubbling	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2008/01/16 15:47
S15	184	S14 and S10	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2008/01/16 15:48
S16	146	S15 and (peroxide or oxide)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2008/01/16 16:25

EAST Search History

S17	578731	(ozone adj oxygen) same (bubble) same fat? oil?	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2008/01/16 16:27
S18	38939	S17 and (water) and produce and peroxide	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2008/01/16 16:28
S19	20795	S18 and emulsion	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2008/01/16 16:29
S20	20587	S19 not S11	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2008/01/16 16:29
S21	2702	ozone same (fat? or oil?)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2008/01/16 16:29
S22	1597	S21 and reaction and water	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2008/01/16 16:30
S23	692	S22 and peroxide	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2008/01/16 16:30

EAST Search History

S24	66	S23 and infection	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2008/01/16 16:31
S25	66	S24 not "I113"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2008/01/16 16:36
S26	19	"274789"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2008/01/16 17:52
S27	2	("0274789").PN.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	OFF	2008/01/16 17:55
S28	4	("3218353").PN.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	OFF	2008/01/16 17:57

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          3972 ORGANICS
        403704 ORGANIC
                 (ORGANIC OR ORGANICS)
       1055059 ORG
         16408 ORGS
       1061039 ORG
                 (ORG OR ORGS)
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       1173617 ORGANIC
                 (ORGANIC OR ORG)
          1426 OZONIDES
        279272 MAKE
        216488 MAKES
        480012 MAKE
                 (MAKE OR MAKES)
        496121 PRODUCE
        183609 PRODUCES
        660972 PRODUCE
                 (PRODUCE OR PRODUCES)
L1           9 ORGANIC (L) OZONIDES (L) (MAKE OR PRODUCE)

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L1 ANSWER 1 OF 9 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 1991:649814 CAPLUS
DOCUMENT NUMBER: 115:249814
TITLE: Aldehydes, hydrogen peroxides, and organic radicals as mediators of ozone toxicity
AUTHOR(S): Pryor, William A.; Church, Daniel F.
CORPORATE SOURCE: Biodyn. Inst., Louisiana State Univ., Baton Rouge, LA, 70803-1800, USA
SOURCE: Free Radical Biology & Medicine (1991), 11(1), 41-6
CODEN: FRBMEH; ISSN: 0891-5849
DOCUMENT TYPE: Journal
LANGUAGE: English

AB It is generally agreed that unsatd. fatty acids (UFA) are an important class of target mols. for reaction with ozone when polluted air is inhaled. Most discussions have implicated the UFA in cell membranes, but lung lining fluids also contain fatty acids that are from 20 to 40% unsatd. Since UFA in lung lining fluids exist in a highly aquated environment, ozonation would be expected to produce aldehydes and hydrogen peroxide, rather than the Criegee ozonide. In agreement with this expectation, the authors find that ozonations of emulsions of fatty acids containing from one to four double bonds give one mole of H_2O_2 for each mole of ozone reacted. Ozonation of oleic acid emulsions and dioleoyl phosphatidylcholine gives similar results, with two moles of aldehydes and one mole of H_2O_2 formed per mol of ozone reacted. The net reaction that occurs when ozone reacts with pulmonary lipids is suggested to be given by the following equation: $R-CH=CH-R' + O_3 + H_2O \rightarrow R-CH=O + R'-CH=O + H_2O_2$. From 5 to 10% yields of Criegee ozonides also appear to be formed. In addition, a direct reaction of unknown mechanism occurs between ozone and UFA in homogeneous org. solution, in homogeneous solns. in water, in aqueous emulsions, and in lipid bilayers to give org. radicals that can be spin trapped. These radicals are suggested to be responsible for initiating lipid peroxidn. of polyunsatd. fatty acids. Thus, aldehydes, hydrogen peroxide, and directly produced org. radicals are suggested to be mediators of ozone-induced pathol.

L1 ANSWER 2 OF 9 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 1991:242254 CAPLUS
DOCUMENT NUMBER: 114:242254
TITLE: The ozonation of unsaturated fatty acids: aldehydes and hydrogen peroxide as products and possible mediators of ozone toxicity
AUTHOR(S): Pryor, William A.; Das, Ballabh; Church, Daniel F.

CORPORATE SOURCE: Biodyn. Inst., Louisiana State Univ., Baton Rouge, LA, 70803-1800, USA
SOURCE: Chemical Research in Toxicology (1991), 4(3), 341-8
CODEN: CRTOEC; ISSN: 0893-228X
DOCUMENT TYPE: Journal
LANGUAGE: English

AB The products of the reactions of ozone with aqueous emulsions of unsatd. fatty acids and with liposomes made from phosphatidylcholine esters were characterized. Ozonolysis of emulsions of Me oleate yields approx. 1 mol of hydrogen peroxide and 2 mol of aldehydes per mol of ozone used and fatty acid reacted. Ozonolysis of emulsions of oleic, linoleic, linolenic, and arachidonic acids gives 1 mol of hydrogen peroxide per mol of ozone used. Only very low yields (less than 5%) of reducible materials other than hydrogen peroxide were observed, suggesting that the yields of org. peroxidic materials, including Criegee ozonides and lipid hydroperoxides, were small. Ozonolysis of rat erythrocyte ghost membranes and rat bronchoalveolar lavage also gives significant yields (about 50%) of hydrogen peroxide based on the moles of ozone consumed. Reactions of ozone with bovine serum albumin, glutathione, and glucose did not produce hydrogen peroxide, implying that the hydrogen peroxide formed during the ozonation of biol. materials arises almost exclusively from ozone/olefin reactions. Hydrogen peroxide and aldehydes are suggested to be important mediators of the modifications observed in both the lung and extrapulmonary tissues when ozone is inhaled.

L1 ANSWER 3 OF 9 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 1974:43746 CAPLUS
DOCUMENT NUMBER: 80:43746
ORIGINAL REFERENCE NO.: 80:7093a,7096a
TITLE: Hydrogenation and ozonolysis of submicrogram quantities of unsaturated organic compounds eluted from gas chromatographic columns
AUTHOR(S): Cronin, D. A.; Gilbert, J.
CORPORATE SOURCE: Procter Dep. Food Leather Sci., Univ. Leeds, Leeds, UK
SOURCE: Journal of Chromatography (1973), 87(2), 387-400
CODEN: JOCRAM; ISSN: 0021-9673
DOCUMENT TYPE: Journal
LANGUAGE: English

AB Procedures are described whereby small amts. of org. compds. are trapped from gas chromatog. columns on short lengths of cooled glass porous layer open tubular capillaries containing either an inert support such as Celite or an adsorbent such as Al2O3. Compds. collected on traps coated with Pd catalyst may be cleanly hydrogenated and the products analyzed by simply breaking the traps in the heated inlet of a gas chromatograph. In a similar manner, ozonolysis may be performed on compds. in uncoated traps, and the resulting ozonides directly cleaved by heat to the carbonyl compds. The cleavage may be carried out in the presence of an added reagent either to form derivs. suitable for gas chromatog., or to produce a characteristic color reaction. The methods outlined are capable of providing structural information on trapped compds. at the 0.05-μg level, and in favorable cases, as low as 0.01 μg.

L1 ANSWER 4 OF 9 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 1966:18812 CAPLUS
DOCUMENT NUMBER: 64:18812
ORIGINAL REFERENCE NO.: 64:3376f-h,3377a-b
TITLE: 1,2,3,4-Butanetetracarboxylic acid and

	1,2,3,4-cyclopentanetetracarboxylic acid
INVENTOR(S):	Volkenburgh, Ross Van; Olechowski, Jerome R.
PATENT ASSIGNEE(S):	Copolymer Rubber and Chemical Corp.
SOURCE:	7 pp.
DOCUMENT TYPE:	Patent
LANGUAGE:	Unavailable
FAMILY ACC. NUM. COUNT:	1
PATENT INFORMATION:	

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 3218353		19651116	US 1962-203433	19620619
PRIORITY APPLN. INFO.:			US	19620619

AB Cyclic olefins were treated with ozone (I) to produce the corresponding stable ozonides in practically quant. yields, and these were decomposed oxidatively to yield the title compds. The ozonides are excellent catalysts for polymerization of ethylenically unsatd. organic compds. A solution of 8.2 g. bicyclo[2.2.1]hept-5-ene-2,3-dicarboxylic anhydride (II) [the Diels-Alder adduct of cyclopentadiene (III) and maleic anhydride], in 100 ml. EtOAc at 5° was treated with a gaseous mixture of O and I containing 4 mole-% I, at a rate of 0.18 mole I/hr. until 1 mole I had been absorbed. The EtOAc was removed in vacuo to give a quant. yield of the corresponding ozonide (IV), m. 135-8° (decomposition). IV was insensitive to shock or impact. Substituting MeOH or AcOH as the solvents in the above reaction as well as in the reaction of the adducts of III and maleic acid yielded a variety of IV derivs. such as the esters and lactones, but the structures which are given for these are presumed and none have been confirmed. cis,cis,cis, cis-1,2,3,4-Cyclopentanetetracarboxylic acid (V), m. 190-1° was prepared by the above procedures. V was converted to the dianhydride, m. 220-2°, by refluxing with Ac_2O. A solution of 9.5 g. II in 35 ml. 90% HCO_2H and 6.8 g. 50% H_2O_2 was refluxed 1.5 hrs. (strongly exothermic reaction), and evaporated to yield 9.5 g. of a carboxylic acid, m. 185-7°, neutralization equivalent 109. A solution of 0.1 mole cis-Δ4-tetrahydrophthalic acid (VI) in 150 ml. EtOAc was ozonized as above and the product decomposed with performic acid to yield dl-1,2,3,4-butanetetracarboxylic acid, m. 227-30°. VI was converted to the ozonide as described above and the product heated to 90° and treated with O for 5 hrs. at the rate of 0.03 cu. ft./min. at 8 psig. to yield meso-1,2,3,4-butanetetracarboxylic acid, m. 189°; tetra-Me ester m. 73-4°. A polymerization mixture containing 70 butadiene, 30 styrene, 5 KOSR flakes, 0.3 trisodium phosphate, 0.1 sulfole, and 180 parts H_2O was charged into 7-oz. polymerization bottles. IV was added in amts. of 0.04, 0.06, and 0.08 parts, resp., and the temperature raised to 60° to yield satisfactory SBR polymer. The other ozonides of this invention were similarly employed. A solution of IV was heated to 90° and oxidatively decomposed with O to yield the dialdehyde derivative of the dicarboxylic acid anhydride, m. 262-3°.

L1 ANSWER 5 OF 9 CAPLUS

ACCESSION NUMBER:	1964:38411 CAPLUS
DOCUMENT NUMBER:	60:38411
ORIGINAL REFERENCE NO.:	60:6744h,6745a
TITLE:	Ozonides
INVENTOR(S):	Hashman, Joseph S.
PATENT ASSIGNEE(S):	Callery Chemical Co.
SOURCE:	1 p.

DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 3115392		19631224	US 1960-73928	19601205
PRIORITY APPLN. INFO.:			US	19601205

AB Me4NO2 and AlBr3 were mixed by a mortar and pestle. Immediate reaction took place and the mixture turned red, qual. indicating ozonides. X-ray diffraction techniques showed the red solid obtained was Me4NO3, tetramethylammonium ozonide. The product was recovered by extraction with liquid NH3. These ozonides are powerful oxidizing agents and can be used to oxidize org. compds. to CO2. Thus, the use of such compds. provides an anal. method whereby the C in org. compds. can be obtained as CO2. The ozonides are also sources of O in self-contained, breathing apparatus where O is not available. The O is released from these compds. upon contact with moisture so that the passage of exhaled air containing moisture through a container of ozonides in a suitable apparatus produces fresh O.

L1 ANSWER 6 OF 9 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 1960:62683 CAPLUS
DOCUMENT NUMBER: 54:62683
ORIGINAL REFERENCE NO.: 54:12111i,12112a-i,12113a
TITLE: Concomitant reaction of elementary sulfur and gaseous ammonia on ketones. XXIII. Synthesis of 1,2,3-trithiolanes and 1,2,4,5-tetrathianes
AUTHOR(S): Asinger, Friedrich; Thiel, Max; Lipfert, Gunter
CORPORATE SOURCE: Univ. Halle-Wittenberg, Germany
SOURCE: Ann. (1959), 627, 195-212
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
OTHER SOURCE(S): CASREACT 54:62683
GI For diagram(s), see printed CA Issue.
AB cf. C.A. 54, 5659i. The action of S and NH3 on ketones produces α-thiol ketones and then Δ3-thiazoline; if H2S is present, substituted 1,2,4,5-tetrathianes result. If NH3 is replaced by an aliphatic primary or secondary (but not tertiary) amine, 1,2,4-trithiolanes are formed. Aldehydes react similarly. The trithiolanes are reversibly cleaved by amines at 0°, and the process shows the function of amines in the synthesis. The cleavage is formulated R2C.S.S.CR2.S .dblharw.R'NH2 R2C(SH)SSC(NHR')R2 .dblharw.R'NH2 R2C(NHR')SSC(NHR')R2 .dblharw.H2S 2 R2C(SH)NHR' .dblharw. 2 R2C:NR' + H2S. At higher temps. this aminolysis is irreversible and produces R2CHSSCHR2 and S, probably by (1) aminolysis of the thioether group (2) displacement of NHR' by SH (3) desulfurization. Reaction of Me2CO with butylammonium polysulfide forms a tetramethyl-2,3,5,6-tetrathiane, probably via BuNHCMe2SSCMe2NHBu, displacement of NHBu by SH, and addition of S to form Me2C.S.S.CMe2S.S. Compds. containing the ring C.S.S.S.C.S.S are also reported. The tetraalkyltrithiolanes (S analogs of ozonides) are quite stable, being unaffected by concentrated aqueous acids, bases, heavy metal salts, and oxidants; KOH in MeOH liberates ketone and H2S, but HCl in MeOH has no effect. Thus, 116 g. Me2CO and 206 g. iso-Bu2NH, saturated at 0° with H2S, treated with 32 g. S, allowed to stand some hrs., acidified, extracted with Et2O, and the extract distilled, gives 80.4 g.

3,3,5,5-tetramethyl-1,2,4-trithiolane, b0.3 32°, nD20 1.546. Similarly are obtained the following 1,2,4-trithiolanes: from 72 g. EtMeCO, 73 g. BuNH2, and 16 g. S, 84 g. 3,5-dimethyl-3,5-diethyl (I), b0.6 70°, nD20 1.542; from 86 g. PrMeCo, 73 g. BuNH2, and 16 g. S, 88 g. 3,5-dimethyl-3,5-dipropyl, b0.3 84°, nD20 1.533, and from 603 g. Et2CO, 365 g. BuNH2, and 123 g. S, 765 g. 3,3,5,5-tetraethyl-, (II), b0.5 95°, nD20 1.541. Similarly, 98 g. cyclohexanone, 73 g. BuNH2, and 16 g. S give 108 g. 3,3,5,5-bis(pentamethylene)-1,2,4-trithiolane (III), m. 50° (EtOH). From aldehydes: 206 g. iso-Bu2NH, saturated at 0° with H2S, 88 g. AcH added slowly followed by 32 g. S, the mixture acidified after 1-2 days, extracted with Et2O, and the extract distilled, gives 7.5 g. 3,5-dimethyl-1,2,4-trithiolane (IIIa), b0.3 38°, nD20 1.597, and 110 g. tarry residue; 116 g. EtCHO, 206 g. iso-Bu2NH, and 32 g. S gives 68.5 g. 3,5-diethyl, b1 77°, nD20 1.567 and 72 g. PrCHO, 120 g. iso-Bu2NH, and 16 g S, 48.5 g. 3,5-dipropyl, b0.4 86°, nD20 1.549, homologs of IIIa. From ketimines and enamines: 74 g. N-(3-pentylidene)butylamine, saturated at 0° with H2S, treated with 8.4 g. S, allowed to come to room temperature in 12 hrs., acidified, extracted with Et2O, and the extract distilled, gives 50.4 g. II; 28 g. 1-morpholinocyclohexene, saturated at 0° with H2S, treated with 2.6 g. S, and distilled, gives 18 g. III. Reduction of 118 g. II with 40 g. LiAlH4 gives 99 g. 3-pentanethiol (IV), b760 112-13°, nD20 1.4459. IV and Me2SO4 gives Me 3-pentyl sulfide, b760 136.5-7.0°, nD20 1.4538; IV and iodine in NaOH gives bis(3-pentyl) disulfide, b0.5 75°, nD20 1.4933; IV and 2,4-dinitrochlorobenzene give 3-pentyl 2,4-dinitrophenyl sulfide, m. 57° (EtOH). Stirring 118 g. II with 146 g. BuNH2 48 hrs. at -20° gives 2 layers; the org. layer and an Et2O extract of the aqueous layer gives on distillation 75 g. N-(3-pentylidene)butylamine, b16 64°, nD20 1.4324, and 32 g. unreacted II; II with BuNH3HS, stirred 25 hrs. at 50-60°, gives bis(3-pentyl) disulfide and bis(3-pentyl) trisulfide, b0.6 96°, nD20 1.5283. Reactions of I are analogous to those of II: reduction with LiAlH4 gives 2-butanethiol; cleavage with BuNH2 gives 40% N-(2-butylidene)-butylamine, b12 45-6°, nD20 1.4286; cleavage with BuNH3HS gives bis(2-butyl) disulfide and bis(2-butyl) trisulfide, b0.5 75-6°, nD20 1.5310. Reaction of 116 g. Me2CO and 206 g. iso-Bu2NH, saturated with H2S, with 64 g. S gives 106 g. 3,3,6,6-tetramethyl-1,2,4,5-tetrathiane, m. 95° (EtOH-Et2O). One 1. cyclohexanone and 700 g. concentrated aqueous (NH4)2S5 on standing 12 hrs. forms crystals; extracting the aqueous layer with Et2O and chilling gives more crystals, total 84 g. 3,3,6,6-bis(pentamethylene)-1,2,4,5-tetrathiane, m. 133° (glacial HOAc). Passage of 6.63 l./hr. NH3 and 4 l./hr. H2S 8 hrs. into 114.7 g. Et2CO and 32 g. S, separation of the org. phase, and fractionation gives II and 3,3,6,6-tetraethyl-1,2,4,5-tetrathiane, b0.5 121°, m. 44-6° (yield not stated), along with unchanged Et2CO and some 2,2,4-triethyl-5-methyl-Δ8-thiazoline. Treatment of 114.7 g. Et2CO and 32 g. S with 6.63 l./hr. NH3 and 7 l./hr. H2S 8 hrs., separation of the org. layer and fractionation, gives 4,4,7,7-tetraethyl-1,2,3,5,6-pentathiepane, b0.5 142-4°, nD21 1.610 (yield not stated). Treatment of 100 g. cyclohexanone 8 hrs. with 1:1 NH3 and H2S, extraction with Et2O, distillation of Et2O and excess cyclohexanone, dilution of the residue with an equal volume of PrOH gives 15 g. 2,2,4,4,6,6-tris(pentamethylene)perhydro-1,3,5-dithiazine, m. 86-7°. Reaction of 32 g. S in 196 g. cyclohexanone with 1:1 H2S and NH3 8 hrs. gives after

extraction with Et2O and distillation, 58 g. oil, b12 126-30°, which crystallizes; the crystals washed with petr. ether give 35 g. 2,2-pentamethylene-4,5-tetramethylene-Δ3-thiazoline, m. 80-1°. The distillation residue, cooled, filtered, and washed with MeOHEt2O, gives 23 g. III.

L1 ANSWER 7 OF 9 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 1945:34125 CAPLUS
DOCUMENT NUMBER: 39:34125
ORIGINAL REFERENCE NO.: 39:5559b-e
TITLE: Heat-convertible, soluble, fusible polymers of divinylbenzene
INVENTOR(S): D'Alelio, Gaetano F.
PATENT ASSIGNEE(S): General Electric Co.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2363836		19441128	US 1942-448158	19420623

AB The parent material for the polymer is the inseparable mixture of 20-30% divinylbenzene (monomer) in diethylbenzene. The monomer is subjected to partial polymerization in the mixture under the influence of heat in the presence of both an inhibitor and a catalyst. The presence of the former permits the retarding of polymerization to a rate at which the formation of the insol. and infusible form of the polymer (a gel) can be readily prevented under industrial conditions. The time required to produce a maximum of the partial polymer is determined by the type and quantity of the inhibitor and catalyst. Partially polymerized divinylbenzene is precipitated from the dialkylbenzene by adding a nonsolvent, e.g., MeOH, EtOH, or glycol. Suitable catalysts are O3, ozonides , inorg., and org. peroxides, and others. The preferred catalyst is Bz2O2 used in quantities of 0.1-2.0% by weight of divinylbenzene. An effective inhibitor is Cu conveniently used in the form of gauze. Among other inhibitors are S, aromatic amines (aniline, diphenylamine, napththylamine, etc.), and hydroxyaryl compds. (phenol, cresol, xylenol, naphthol, etc.).

L1 ANSWER 8 OF 9 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 1917:15035 CAPLUS
DOCUMENT NUMBER: 11:15035
ORIGINAL REFERENCE NO.: 11:3029g-i,3030a-i,3031a-c
TITLE: Refractometric researches on the hydrocarbons with two and three conjugated double bonds. IV. The formenic terpenes and their derivatives. Synthesis of two new 1,3,5-trienes
AUTHOR(S): Enklaar, C. J.
CORPORATE SOURCE: La Haye
SOURCE: Recueil des Travaux Chimiques des Pays-Bas et de la Belgique (1916), 36, 215-46
CODEN: RTCPB4; ISSN: 0370-7539
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
GI For diagram(s), see printed CA Issue.
AB The paper is divided into six subheadings: I. The constitutional formulas of ocimene and of alloocimene. E. has previously reported on the

ozonization of ocimene, dihydroocimene and alloocimene (C. A. 1, 2117; 2, 2800; 3, 1010) as carried out in Harries' laboratory. These expts. have now been amplified. The alloocimene (a) was ozonized with 5-8% O3 and gave an ozonide that is nearly completely soluble in H2O. This solution gives only traces of a product that reacts with PhNHNH2, giving a compound, m. 230° (no trioxymethylene was formed). It was seen that the ozonide thus decomposing abnormally was obtained by the action of higher concns. of O3. The ozonide of dihydroalloocimene (b) decomposed in H2O gave products that react feebly for pyrrole; the presence of Me2CO could not be proved, but an unidentified aldehyde or ketone, b. about 80°, was found. Levulinic aldehyde, found with dihydroocimene treated similarly, was absent. The ozonides of ocimene in aqueous solution gave pyrrole with Zn dust. E. concludes from these data that (a) contains the group Me2C:CH. This group is not present in (b) because Me2CO is not obtained from its ozonide. Considering that trioxymethylene is not formed the provisional formula of (a) becomes Me2C:CH.CH:CHCMe:CHMe. The formula for ocimene is H2C:CMeCH2CH2CH:CMeCH:CH2 since only 0.5 as much Me2CO was obtained as with (a). Thus ocimene and alloocimene are mixts. of compds. having the following partial formulas: CH2:CMeCH2(C6H9) and Me2C:CH(C6H9) in unequal amts. Moreover the low boiling fractions of (a) contain a stereoisomer of ocimene as shown by new expts. E. considers that the presence of malonic aldehyde and acid as oxidation products of ocimene is not fully proved as shown by work now under way. The compound that m. 230° resembles diacetylosazone both in composition and m. p. The decomposition of the ozonide of (a) would thus resemble that of citronellal:....CH.CMe.CHMe.fwdharw....CHO + MeCH(OH)COMe, giving the unknown butanolone and diacetylosazone (c) with PhNHNH2. The confirmatory experiment of preparing (c) from octatriene could not be carried out for the lack of material. That the osazone may be that of fumaric aldehyde is briefly discussed. Priority claims against Auwers and Eisenlohr (C. A. 6, 72) are discussed. II. The refractometric constants of the compound having the formula Me2C:CHOH:CHCMe:CHMe, deduced from observations on the new synthetic trienes, octatriene and nonatriene. Since the proposed formula for (a) was deduced in 1909, E. has been trying syntheses, the results of which are described below. Two new hydrocarbons were obtained besides two homologs of vinylallyl-carbinol by a method previously described (C. A. 7, 1169, 1074; 10, 904). 53 g. tiglic aldehyde (Grignard, Abelmann, C. A. 4, 2459) were obtained from 125 g. PrH + 150 g. AcH. The product was impure as shown by careful refractometric detns. 26 g. tiglic aldehyde, b119 63.2-65°, with 36.5 g. allyl bromide, b762 69.3-70°, in an equal volume of dry Et2O added slowly to 58.6 g. Zn separated 9.1 g. excess Zn on adding 1 volume Et2O. The reaction products were dried with CaCl2 and fractionally distilled The first fraction b21 78-9° is nearly pure 5-methyl-1,5-heptadien-4-ol (d), CH2:CHCH2CH(OH)CMe:CHMe; yield about 50%. Pure (d) b760 172-3°, has a fresh odor resembling menthol, absorbs O2 rapidly, d412.2 0.8766; na 1.46174 and nD 1.46481 at 11.8°; the calculated and observed values for Mα are 39.490 and 39.508, resp., and 39.733 and 39.735, resp., for MD 15 g. (d) mixed carefully with 25 g. KHSO4 and heated on a sand bath under 70-100 mm. pressure in a current of CO2 gave 4 g. of a hydrocarbon which after washing with NaOH, drying with CaCl2 and distilling under reduced pressure in the presence of liquid Na + K alloy gave 3 fractions of the octatriene (e), under 96 mm., totalling 3.35 g., of which the physical consts. were determined (e) is unstable in the presence of acids: 2% H2SO4 in glac. AcOH polymerizes it at 40° in less than 1 min. Hydrogenation of (e) with EtOH + Na was very unsatisfactory. 2 atoms Br were quickly absorbed by (e) at -20° and the product slowly absorbs 2 more: the oil obtained smells like

camphor. Heated with Na it gives a viscid transparent polymer soluble in C6H6 from which a white powder is precipitated by EtOH. (e) absorbs O2 very rapidly: NH3-Ag2O is not reduced but HgCl2 in EtOH is quickly reduced to HgCl. Qual. expts. suggested that (e) like cyclopentadiene gives an isonitrosylic compound with EtNO2 + EtONa in EtOH. α,β-Methylethylacrolein was prepared (Grignard, Abelmann, l. c.) and treated as above gave 5-methyl-1,5-octadien-4-ol (f), H2C:CHCH2CH(OH)CMe:CHCH2Me, in 3 fractions of which the physical consts. were determined (f) treated with KHSO4 as with (d) gave the nonatriene, b36 85-7° when boiled under 760 mm. it polymerizes: the chemical properties seem to resemble those of (e). In discussing the refractometric data, E. concludes that the impurities present prevent any sharp determination of values. These impurities are thought to be polymers rather than stereoisomers. In general the results indicate that these hydrocarbons give exaltations greater than those of the aromatic series: with the trienes the difference is proportionally greater than with dienes. III. Refractometric constants of alloocimene. The refractometric consts. of alloocimene prepared in various ways are discussed comparatively. IV. Refractometric constants of methylated butadienes. V. Refractometric constants of ocimene. The comparison of the refractometric values of ocimene and those of methylated butadienes shows that they agree with those that a hydrocarbon having a system of double bonds interrupted at the center should have. VI. Refractometric constants of 2,6-dimethyloctane. The presence of impurities in ocimene would be disclosed by the physical properties of decane obtained from it by hydrogenation with active Ni according to Sabatier and Senderens. The same decane was studied by Eykman (Chemical Weekbl. 3, 653(1906)) and obtained by hydrogenating geraniol. The agreement in the consts. for the decane from these 2 sources proves the purity of ocimene again. In a supplement, E. describes a modified technic for the combustion of volatile and difficultly combustible organic compds. in order to obtain accurate analytical data. The modification depends on the use of PbCrO4 instead of CuO and is designed to make its use safe and certain under the above conditions. For the large amount of numerical details in this paper see the original.

L1 ANSWER 9 OF 9 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 1911:20522 CAPLUS
DOCUMENT NUMBER: 5:20522
ORIGINAL REFERENCE NO.: 5:3519a-i,3520a-i,3521a-i,3522a-f
TITLE: Hydrocarbons of the Butadiene Series and Artificial Caoutchoucs Prepared from them
AUTHOR(S): Harries, C.
CORPORATE SOURCE: Chem. Inst., Univ. Kiel
SOURCE: Ann. (1911), 383, 157-227
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
GI For diagram(s), see printed CA Issue.
AB Methyltetramethyleneglycol, HOCH2CH2CHMeCH2OH, is prepared by adding di-Et pyrotartrate (propane-α,β-dicarboxylate), in absolute alc., to Na, the mixture is shaken vigorously and subsequently heated. After removal of the alc., the solid is dissolved in H2O, decompose by CO2 and the filtrate concentrate at 80°. in a vacuum. Colorless liquid resembling glycerol, b13 124-5°; d1818 0.9954; nD18 1.45173; nα18 1.44925; nγ18 1.46107; MD 28.20; Mγ-Mα 0.656. Yield, 20% of the ester. Diacetyl derivative, C9H16O4, from the glycol and b. Ac2O; water-clear, mobil liquid with an odor of mustard, b17 116-7°; d2020 1.0434; nD20 1.42717; nαD 1.42509; nγ20 1.43658; MD 46.3; Mγ-α, 1.08. Yield, about 150% of the

glycol. Sym.-diphenyldiurethan, $C_{19}H_{22}O_4N_2$, from the glycol and Ph isocyanate; lustrous plates from ligroin (b. p. 70-100°), m. 97°. Yield, 120% of the glycol. Tetramethyldiurethan, $C_{31}H_{30}O_4N_2$, from the glycol and diphenylcarbamyl chloride, in presence of pyridine; colorless prisms from ligroin, m. 102°. Yield, 180° of the glycol. β-Methyltetramethylene oxide formula (I) below, is prepared from the glycol and H_2SO_4 (60%), at 100°; colorless, mobil liquid with a pleasant, strongly ethereal odor, b. 86-7°; d2020 0.8643; nD20 1.41122; nα20 1.40905; nγ20 1.41979; MD 24.74; Mγ-Mα 0.577. Yield, 40-50% of the glycol. β-Methyltetramethylene bromide, $BrCH_2CH_2CHMeCH_2Br$, from the glycol and concentrate HBr, at 100°; heavy, colorless liquid with a sweet odor, b11 84-6°; d1717 1.6986; nD17 1.51217; nα17 1.50918; nγ17 1.52722; MD 40.64; Mγ-Mα 1.19. Yield, 190% of the glycol. On exposure to light the bromide evolves HBr; when distilled with quinoline (3.75 pts.) it gives isoprene containing some dimethylene. Yield, poor. A better method of obtaining isoprene consists in treating this dibromide with Me_3N, in absolute alc., at 100°, and shaking the viscid product with moist Ag_2O, after filtering and concentrate under reduced pressure, the liquid is distilled under the ordinary pressure. The resulting Me_3N is absorbed by H_2O and the isoprene purified by dilute H_2SO_4, followed by Na. It had the following properties: b. 36-7°; iD 51°; iα 51°; nD 1.42267; nα 1.41807; iγ 52° 5'; nγ 1.44340 all at 21°; d2121 0.6804; d421 0.6793; MD 25.45; Mγ-Mα 1.33; ΣD 37.46; EΣD 1.72 (4.8%); Σγ-α 1.958; EΣγ-α 0.572 (41.3%). Yield, 50%. The yield of dimethylethylcarbinol from acetone and EtBr is about 80%. The carbinol is volatil with Et_2O; with anhydrous $C_2H_2O_4$ (0.5 mol.), or H_2SO_4 it gives trimethylethylene. Yield, above 70%. In glacial AcOH, Br converts it into the dibromide; b30 70°. Yield, of pure compound, about 230% of the hydrocarbon. The preparation of isoprene from trimethylene dibromide is best accomplished by dropping it on well dried soda lime, which has been saturated with CO_2; the temperature inside the apparatus should be 600°. The decompose is carried out in a vessel of special design; it is made of Fe or brass and the soda-lime is divided by wire gauze, so as to prevent the $CaBr_2$, which is formed, from clogging. Yield, of isoprene, 18-20% of the bromide. Omission of the CO_2 produces an impure product, which probably contains trimethylethylene. When trimethylethylene dibromide is dropped into fused KOH, the product consists of a little hydrocarbon together with an unsaturated, monobromo compound, from which the Br is removed only with difficulty. Butadiene (erythrene) is best prepared, in a state of purity, by the exhaustive methylation of N-methylpyrrolidone (Ciamician and Magnaghi). The process described in Ger. Pat. 28,390 IV., 39, starting from p-cresol, did not give good yields under laboratory conditions. The best results were obtained as follows: com. methylethyl ketone, in Et_2O, was reduced by H_2O and Na. The resulting sec. butyl alc. is volatil with Et_2O. Yield, 73%. It is converted into butylene by heating with P_4O_{10}. Yield, about 47% of the alc. The use of H_2SO_4, or of $C_2H_2O_4$ gives a less pure product. With well cooled $CHCl_3$ and Br, it forms butylene bromide, b12 62°. Yield, about 120% of the butylene. It was converted into butadiene by means of soda-lime, in the manner described above. The product is purified by conversion into the tetrabromide, by means of Br, in $CHCl_3$, at the temperature of ice + NaCl. Yield, 22%. β,γ-Dimethylbutadiene, $H_2C:CMeCMe:CH_2$, is best prepared by converting pinacone into the hydrochloride and decomposing this by means of soda-lime, in the manner described above. The product b. 71°; d416.5 0.7304; nD 1.44321; nα 1.43870; nγ 1.46303, all at 16.5°; MD

29.80; $M\gamma$-$M\alpha$ 1.41. The best method of comparing natural and artificial caoutchoucs consists in an examination of the ozonides and of their decomposition products. The other possible derivatives are the nitrosites and the bromides, which are amorphous, have no definit m. ps. and can only be purified with great difficulty, if at all. Isoprene is converted into isoprene caoutchouc, (C10H16)x, by heating under pressure, with an equal volume of glacial AcOH, on a H2O bath, during 8 dys. The yield varies widely and it is but little, if at all affected by marked variation in the proportion of AcOH, or by changes in the temperature (95-120°). When only a few drops of AcOH are employed, it is necessary to distil the product under the ordinary pressure to remove isoprene and its dimol. polymerization compound, then at 105° (10 mm.) to remove an oily material. The residue is transparent, almost colorless and closely resembles natural caoutchouc. When recently prepared it is readily soluble in ordinary organic media, but this property gradually changes and finally the material becomes insol. After repeated precipitation, natural caoutchouc exists in 3 modifications, the (a) form is represented by ordinary, solid caoutchouc, the (b) form is quite insol. and the (c) modification is oily. All are mutually transformable, (c) into (a) slowly and spontaneously; (a) into (c) by warming in solution; (a) into (b) slowly and spontaneously; (b) into (a), or (c) by b. with AcOH, or Ac2O. Similar modifications are observed in the case of artificial caoutchouc, the purer it is the more easily does it pass into the (b) form, but the reverse transformation has not been realized. The best yield of artificial caoutchouc is obtained from the purest specimens of isoprene and this is true irrespective of the method used for the preparation of the isoprene. Artificial caoutchouc, like the natural product, after drying, must be retained in an atmospheric of CO2 and must be protected from light, otherwise it soon becomes brittle. The formula, (C10H16)x, only applies to artificial caoutchouc which has been prepared in an atmospheric free from O. Specimens formed in contact with air contain O, as also does natural caoutchouc and it is probable that the (b) form mentioned above is "ordinary" caoutchouc which has become coated with the oxidation product. This latter is supposed to be impervious to ordinary, organic, liquid media and, consequently, the unchanged caoutchouc cannot dissolve in them. [NOTE. For some reason, not stated, the author speaks of the formation of this substance as "autooxidation" and refers to the product as the "autooxidation product." His formulas for isoprene, for its polymerization compound and for the varieties of pure caoutchouc, show, however, that these substances are free from O. ABSTR.] Comparative vulcanizing expts. (cold process) indicate that probably the best specimens of isoprene caoutchouc are produced when polymerization takes place at the lowest possible temperature and without the addition of any foreign substance. A comparison of the "tetrabromides" of natural and artificial caoutchouc shows that the latter is more readily soluble in CS2; other slight differences are also mentioned. In both cases different specimens of the bromides vary in comp. and it is difficult to prepare from either variety of caoutchouc a bromo derivative of the formula, C10H16Br4. The nitrosite (c), (C10H15O7N3)2, (decompose 158-62°) from natural caoutchouc has been described previously. It is also formed from isoprene caoutchouc, but with somewhat greater difficulty. Both varieties of caoutchouc agree in their behavior towards O3 (12%). The diozonide, C10H16O6, which is formed, when decompose with hot H2O, gives levulic acid (3.2 g.), levulic aldehyde (1.5 g.), resin (0.4 g.), loss (2.2 g.). These yields were from 7 g. of ozonide; they vary somewhat in different expts. There are indications that isoprene caoutchouc contains an isomeric caoutchouc. The distillation of natural caoutchouc (0.1-0.2

mm.) has been described by E. Fischer and C. Harries (Ber., 35, 2162 (1902)). Exactly similar results have been obtained with isoprene caoutchouc. Other substances, besides caoutchouc, are formed by the polymerization of isoprene and several of them have been described by other chemists. The author has isolated from the mixture a hydrocarbon, $C_{10}H_{16}$; b14 63-5°; d1818 0.8451; nD 1.47408; nα 1.47113; nγ 1.48860, all at 18°. It contains but little dipentene. Yield, variable. Ozonide, oily. Butadiene caoutchouc, $(C_8H_{12})_\chi$, is prepared from butadiene alone, in a vacuum at 100-10°, or with glacial AcOH, at 110-20°, during 10 dys. in each case. The product resembles closely that from isoprene and similar precautions regarding its oxidation are necessary. At 35°, in presence of Na, butadiene is converted into a new, highly insol. variety of caoutchouc which does not react with HNO_2, or with O_3. Butadiene caoutchouc, prepared in presence of glacial AcOH, when treated with HNO_2 in excess, gives a yellow, amorphous nitrosite, decompose above 80° (gas evolution). A terpene, C_8H_{12}, is formed together with butadiene caoutchouc; b23 36°; d416 0.8523; nD 1.46768; nα 1.46423; nγ 1.48812, all at 16°. In concentrate H_2SO_4 the color is yellow, in alc. H_2SO_4 reddish yellow. Ozonide, white and sparingly soluble; it is rather explosive and when b. with H_2O forms an aldehyde which gives the pyrrole reaction. Dimethylbutadiene is best polymerized to dimethylbutadiene caoutchouc, $(C_{12}H_{20})_\chi$, by heating it without a solvent, at 100°, during about 23 dys. The product is distilled in a vacuum (up to 110°); the residual caoutchouc resembles closely that from isoprene, but is somewhat more readily soluble Yield, 32% of the dimethylbutadiene. Bromide, $C_{12}H_{20}Br_4$, gray and amorphous. It dissolves with difficulty in CS_2, evolves HBr about 130° and blackens at higher temps. Yield, not quant. Nitrosite, $C_{12}H_{19}O_7N_3$, yellow and amorphous, becomes brown 120°, not decompose 200°. Yield, quant. Diozonide, $C_{12}H_{20}O_6$, is prepared in CCl_4, during 9 hrs.; thick, clear liquid from AcOEt + petroleum ether. It gives acetonylacetone when decompose by b. with glacial AcOH. A second ozonide is formed, together with the preceding compound and is separated by its soluble in AcOEt + petroleum ether. When decompose as above it gives a mixture of substances containing acetonylacetone. The preparation of a caoutchouc, $(C_8H_{12})_\chi$, by the action of Na on butadiene is described above. The substance will be referred to in this abstract as "butadiene caoutchouc Na" and its analogs will be designated in a similar manner. After the completion of the polymerization the Na is removed by means of dilute alc.; the product is light yellow, its solubility gradually decreases, it may be vulcanized very readily and the resulting material is superior to any other artificial caoutchouc in elasticity and flexibility. Yield, almost quant. In $CHCl_3$, with O_3 (about 6-7%), it gives a diozonide, $C_8H_{12}O_6$; white solid, which is also said to be an oil. It is readily soluble, but is difficult to purify. With O_3 (12-14%) the product consists chiefly of a mono-ozonide, probably $C_8H_{12}O_3$; it is insol. in $CHCl_3$, AcOEt, or glacial AcOH. The primary product with O_3 of the higher concentrate appears to be an ozoneoxide, $C_8H_{12}O_4$, which is solid and highly explosive. In Et_2O it changes to the monoozonide. Decompose of these ozonides gives substances which were not identified, except that H_2O_2 is formed from the diozonide. Nitrosite, yellow, amorphous, insol. and of variable composition. Bromide, white powder from CS_2 + ligroin. It appears to be a mixture of $C_8H_{12}Br_2$ and $C_8H_{12}Br_4$. Yield, variable. Isoprene caoutchouc Na, $C_{10}H_{16}$, is prepared from pure isoprene and Na wire (0.02-5 pts.), at 60°, during 50 hrs. and is purified in the manner described above. Yield, practically quant. It resembles isoprene caoutchouc in general properties, but dissolves more easily when recently prepared and may be vulcanized very readily. In $CHCl_3$, with O_3, (12-4%),

during 10 hrs., it gives a diozonide, C10H16O6; white solid from AcOEt + petroleum ether, it may be powdered; mol. weight, in glacial AcOH, 250-350. By the use of CCl4, in place of CHCl3, a mixture of mono- and diozonides is formed; they were not separated completely. When decompose with H2O, or glacial AcOH the ozonides give a trace of levulic aldehyde, together with a brown oil; it decompose when distilled and exhibits the pyrrole test only faintly. With HNO2 the caoutchouc forms 2 nitrosites, they are separated by means of acetone. The soluble compound, C10H16O5N2, which is formed in much the larger quantity, is a pale yellow powder, darkens 170°, not m. 260°. The nitrosite insol. in acetone does not appear to have been analyzed. Total yield, 200% of the caoutchouc. Bromide, C10H16Br4, white powder from CS2 + ligroin. It resembles isoprene caoutchouc tetrabromide. Yield, 200% of the caoutchouc. Dimethylbutadiene caoutchouc Na, (C12H20)χ, is prepared in a similar manner to isoprene caoutchouc Na, at 60°, during 240-88 hrs.; it is very different from dimethylbutadiene caoutchouc and resembles gutta-percha in appearance. Ozonide, thick oil, soluble in CHCl3. Yield, 66% of the caoutchouc. When decompose it gives H2O2, a little acetonylacetone and a relatively large quantity of substances which could not be identified. This shows that the dimethylbutadiene caoutchouc Na contains some dimethylbutadiene caoutchouc. Nitrosite, C12H19O7N3, from AcOEt + petroleum ether. It dissolves readily in Et2O. Yield, 227% of the caoutchouc. Tetrabromide, C12H20Br4, white powder from CS2 + ligroin, evolves HBr 130°, not m. 200°. Yield, 150% of the caoutchouc. These results especially those obtained by the decompose of the ozonides, show that the caoutchoucs obtained by the use of Na are different from those prepared by direct polymerization. The paper closes with a discussion of the structure of caoutchouc and a reply to Pickles (C. A., 4, 2936). The author still considers that the basic hydrocarbon of natural caoutchouc is 1,5-dimethylcyclo-1,5-octadiene (II). Tilden was, of course, the first to obtain artificial caoutchouc.

```
=> d his

     (FILE 'HOME' ENTERED AT 16:51:32 ON 16 JAN 2008)

     FILE 'CAPLUS' ENTERED AT 16:51:59 ON 16 JAN 2008
L1            9 S ORGANIC (L) OZONIDES (L) (MAKE OR PRODUCE)

=> s ozonized (L) (fat? or oil?) (L) water
          5658 OZONIZED
        763299 FAT?
        939411 OIL?
       2650614 WATER
        271094 WATERS
       2708560 WATER
                 (WATER OR WATERS)
L2         203 OZONIZED (L) (FAT? OR OIL?) (L) WATER

=> l2 and (produce or make) (L) peroxide
L2 IS NOT A RECOGNIZED COMMAND
The previous command name entered was not recognized by the system.
For a list of commands available to you in the current file, enter
"HELP COMMANDS" at an arrow prompt (=>).

=> s l2 and (produce or make) (L) peroxice
        496121 PRODUCE
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        183609 PRODUCES
        660972 PRODUCE
                 (PRODUCE OR PRODUCES)
        279272 MAKE
        216488 MAKES
        480012 MAKE
                 (MAKE OR MAKES)
             0 PEROXICE
             2 PEROXICES
             2 PEROXICE
                 (PEROXICE OR PEROXICES)
             0 (PRODUCE OR MAKE) (L) PEROXICE
L3           0 L2 AND (PRODUCE OR MAKE) (L) PEROXICE

=> s l2 and (produce or make) (L) peroxide
        496121 PRODUCE
        183609 PRODUCES
        660972 PRODUCE
                 (PRODUCE OR PRODUCES)
        279272 MAKE
        216488 MAKES
        480012 MAKE
                 (MAKE OR MAKES)
        225166 PEROXIDE
         48785 PEROXIDES
        244405 PEROXIDE
                 (PEROXIDE OR PEROXIDES)
          6650 (PRODUCE OR MAKE) (L) PEROXIDE
L4           3 L2 AND (PRODUCE OR MAKE) (L) PEROXIDE

=> d l4 1-3 ibib abs
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L4 ANSWER 1 OF 3 CAPLUS

ACCESSION NUMBER: 1963:80967 CAPLUS
DOCUMENT NUMBER: 58:80967
ORIGINAL REFERENCE NO.: 58:13770c-h,13771a-h,13772a-h
TITLE: Double-bond isomerization in higher-molecular olefins. IV. The bond-isomerizing effect of various compounds on n-undecenes
AUTHOR(S): Asinger, Friedrich; Fell, Bernhard; Collin, Gerd
CORPORATE SOURCE: Tech. Hochschule, Aachen, Germany
SOURCE: Chemische Berichte (1963), 96, 716-35
CODEN: CHBEAM; ISSN: 0009-2940
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

AB cf. CA 37, 46841,2·; 50, 7047f. The double-bond isomerization in 1-(I) and 5-undecene (II) by acidic, basic, and neutral compds. was investigated. A particularly effective isomerization catalyst is Fe(CO)5 (III) decomposing thermally or under ultraviolet irradiation. The quant. analysis of the isomeric n-undecenes was performed by oxidative ozonolysis and gas chromatography of the resulting carboxylic acids as their Me esters. CH2:CHCH2Br (266 g.) in 800 cc. dry Et2O added dropwise with stirring and cooling to C8H17MgCl from 60 g. Mg and 327 g. C8H17Cl in 1200 cc. dry Et2O, refluxed 2 hrs. with stirring, poured onto 1-2 kg. crushed ice, and worked up yielded I, b13 74.5-6.0°, b755 191.5-2.5° n20D 1.4262, containing 0.2% II, 2.7% 2-undecene (IV), 0.8% 3-undecene (V), and 0.2% 4-undecene (VI). A series of 4 similar runs was performed to yield I of 93.1, 96.3, 97.1, and 95.1% purity, resp. AmOH with SOCl2 in

C5H5N yielded 30% AmCl, b736 107-7.7°, n20D 1.4128-1.4129. AmCHO (101 g.) in 200 cc. dry Et2O added dropwise to AmMgCl from 118 g. AmCl and 29 g. Mg in 600 cc. dry Et2O, poured onto 250 g. ice, treated with 200 cc. dilute HCl, and worked up yielded 117 g. Am2CHOH (VII), b12 112.5-12.8°, n25D 1.4345, m. 24.8-5.8°; 2,5-dinitrobenzoate m. 48°. VII (205 g.) added dropwise during 0.5 hr. at 100° to 333 g. C17H35COCl, stirred 3 hrs. at 120°, cooled to 80°, treated with 5 cc. MeOH, heated slowly to 290° while being treated with N and then to 320°, and the distillate fractionated yielded 125 g. II, b724 190.5-1.5°, n20D 1.4293, containing 1.5% VI. The isomeric undecenes (2 g.) in 25 cc. hexane ozonized about 15 min. at -15° with about 25 l. O containing 4 volume-% ozone/hr., added at 90° to 15 g. Ag2O in 70 cc. 10% aqueous NaOH during 10 min. with stirring, stirred 3 hrs. on the water bath, cooled to 50°, treated dropwise with stirring during 5 min. with 43 cc. 65% HNO3, cooled to room temperature, and extracted with pentane, and the extract concentrated
gave the carboxylic acids; the crude product dissolved in 100 cc. 4% KOH in 1:1 MeOH-H2O, washed with C6H6 and petr. ether, the washings reextd. with 4% KOH in 1:1 MeOH-H2O, the combined alkaline exts. acidified with 65% HNO3 and extracted with pentane, and the pentane extract treated with CH2N2 from
about 3 g. H2NCON(NO)Me in pentane gave the corresponding Me esters which were gas-chromatographed. The compds. measured were C9H19CO2Me, C8H17CO2Me, C7H15CO2Me, C6H13CO2Me, and AmCO2Me for I, IV, V, VI, and II, resp. I (3.0 g.) in 35 cc. pentane ozonized at -15° during 1 hr. under the usual conditions and worked up showed that the lower homologs in the resulting acid mixture were enriched only by about 3.5%. Ozonization at -60° (performed in the same manner) resulted in incomplete reaction. I (4.5 g.) and 0.29 g. Li, or 1.0 g. Na, or 1.7 g. K refluxed with stirring gave a suspension of the alkali metal; the isomerization runs with the alkali metals were carried out with 40 mole-% I and 60 g.-atom % alkali metal in the absence of an added halo compound and with 38 mole-% I and 57 g.-atom % metal in the presence of 5 mole-% halo compound; after completion of the run, the mixture was filtered, and the filtrate distilled to give the isomer mixture which was analyzed in the usual manner. By this procedure were performed the following runs (catalyst, added halo compound, reaction time in hrs., % purity of I used, and % content of I, IV, V, VI, and II in the resulting isomer mixture given): Li, none, 5, 96.3, 91.8, 5.1, 1.1, 1.1, 0.9; Li, C8H17Cl, 5, 96.3, 92.1, 5.6, 1.1, 0.6, 0.5; Li, PhCl, 2, 96.3, 92.6, 5.2, 1.0, 0.6, 0.5; Na, none, 5, 93.4, 90.2, 8.2, 1.0, 0.4, 0.3; Na, C8H17Cl (1.8 mole %), 5, 95.3, 90.4, 7.3, 1.1, 0.7, 0.5; Na, C8H17Cl (5 mole %), 5, 93.1, 67.0, 30.4, 1.6, 0.6, 0.4; Na, PhCl, 5, 96.3, 2.7, 32.2, 29.1, 24.0, 12.0; Na, PhCl, 0.5, 96.3, 2.1, 31.5, 28.5, 24.9, 13.0; Na, PhBr, 5, 96.3, 4.5, 32.4, 27.2, 22.9, 13.1; Na, o-MeC6H4Cl, 5, 96.3, 1.3, 28.0, 26.6, 26.9, 17.2; Na, m-MeC6H4Cl, 5, 96.3, 1.5, 28.3, 26.4, 27.0, 16.8; Na, CH2:CHCH2Cl, 2, 96.3, 93.9, 4.8, 0.7, 0.3, 0.3; Na, p-MeC6H4Cl, 5, 96.3, 3.6, 31.8, 27.3, 23.6, 13.6; K, none, 0.5, 96.3, 91.8, 6.2, 1.2, 0.5, 0.3; K, C8H17Cl, 0.5, 96.3, 89.3, 6.9, 1.4, 1.7, 0.7; K, PhCl, 0.5, 96.3, 13.1, 65.8, 16.5, 3.6, 1.1. A similar run with 5 mole-% 0.7M PhLi refluxed 5 hrs. under Ar (after removal of the Et2O) gave from 97.1%-pure I a mixture containing 92.2 I, 1.5 IV, 0.4 V, 0.4 VI, and 0.4% II. I (3.0 g.) (97.1%-pure) refluxed 5 hrs. with 5 mole % PhLi in 20 cc. dry Et2O and decomposed with H2O, and the organic phase worked up yielded a mixture containing 97.2 I, 1.8 IV, 0.3 V, 0.3 VI,
and 0.4% II. A run with 97.1%-pure I and 50% BuLi at 25° under Ar during 24 hrs. gave a mixture of 96.7 I, 2.4 IV, 0.5 V, 0.4% VI, and a trace

of II. Na (about 3 g.) as 10 cc. 33% Na dust dispersion in xylene, 30 cc. MePh, and 5.5 g. dry PhCl stirred and treated with pure Ar during 1 hr. at 35-40°, the mixture treated with 7.7 g. 97.1%-pure I, stirred 1 hr. at 50° under Ar, poured onto powdered dry ice in portions, diluted with H2O, and acidified gave 5.7 g. BzOH; the organic phase dried and distilled gave a mixture containing 3.8 I, 85.6 IV, 8.5 V, 1.4 VI, and 0.7% II. A run with II performed during 0.5 hr. with Na and PhCl in the usual manner gave a mixture containing 0.2 I, 4.2 IV, 11.4 V, 36.7 VI, and 47.5% II. All alkali alcoholates used for the isomerization runs were obtained by dissolving the alkali metal in a large excess of the appropriate alc., mixing an aliquot containing 5 mole-% and adding to the I and then removing the excess alc. with the exception of C8H17OH. (BuO)2Ca was obtained similarly by refluxing Ca in BuOH for 12 hrs. B(OAc)8 from B(OH)3 and Ac2O transesterified with the equivalent amount appropriate alc. yielded the corresponding B(OR)3 (R, % yield, b.p./mm., and n20D given): Pr (VIII), 22, 74-84°/13, 1.3954; Bu (IX), 40, 109-14°/13, 1.4094; iso-Bu (X), 38, 105-10°/19, 1.4033; EtMeCH (XI), 32, 88-95°/19, 1.3972; Am (XII), 48, 146-51°/16, 1.4190. TiCl4 treated with NaOEt in absolute EtOH yielded 10% Ti(OEt)4 (XIII), b13 154-4.5°. I (3.0 g.) and 5 mole-% appropriate alcoholate or ester refluxed 5 hrs. at 190° and distilled gave the isomer mixture A series of runs was performed in this manner (alcoholate or ester used as catalyst, % purity of I used, and % content of I, IV, V, VI, and II in the resulting isomer mixture given): BuOLi, 96.3, 94.8, 4.0, 0.7, 0.3, 0.2; EtMeCHOLi, 96.3, 96.8, 2.4, 0.4, 0.3, 0.2; tert-BuOLi, 96.3, 96.7, 2.6, 0.3, 0.3, 0.1; C8H17OLi, 96.3, 93.8, 2.8, 2.8, 0.5, 0.1; BuONa, 96.3, 95.8, 3.1, 0.6, 0.3, 0.2; EtMeCHONa, 96.3, 96.4, 2.2, 0.6, 0.3, 0.5; tert-BuONa, 96.3, 96.9, 2.0, 0.4, 0.3, 0.4; C8H17ONa, 93.1, 90.2, 8.2, 1.0, 0.4, 0.3; BuOK, 96.3, 78.0, 17.9, 2.7, 0.9, 0.4; EtMeCHOK, 96.3, 42.5, 54.7, 1.8, 0.6, 0.3; EtMeCHOK, 96.3, 47.1, 49.7, 2.2, 0.7, 0.4; EtMeCHOK, 96.3, 62.2, 36.1, 1.2, 0.4, 0.2; tert-BuOK, 96.3, 72.7, 24.1, 1.7, 1.0, 0.6; tert-BuOK (in tert-BuOH), 96.3, 74.2, 21.9, 2.4, 0.9, 0.6; C8H17OK, 96.3, 93.5, 4.8, 1.2, 0.3, 0.2; C8H17OK, 96.3, 91.5, 6.4, 1.6, 0.3, 0.2; C8H17OK, 96.3, 89.9, 6.7, 1.9, 0.9, 0.4; 20% KOH-(CH2OH)2 (25 mole %) (at 180°), 96.3, 94.3, 3.8, 0.8, 0.4, 0.6; (BuO)2Ca, 96.3, 93.6, 4.4, 1.0, 0.5, 0.5; (iso-PrO)3Al, 93.1, 91.9, 5.4, 2.2, 0.3, 0.2; (EtMeCHO)3Al, 93.1, 92.8, 5.8, 0.9, 0.3, 0.2; VIII, 93.1, 90.9, 6.8, 1.5, 0.6, 0.3; IX, 93.1, 91.3, 6.7, 1.2, 0.4, 0.3; X, 93.1, 91.3, 6.7, 1.2, 0.4, 0.3; XI, 93.1, 92.4, 5.7, 1.2, 0.5, 0.3; XII, 93.1, 91.7, 6.2, 1.4, 0.5, 0.3; XIII, 93.1, 93.1, 5.3, 1.0, 0.4, 0.2; (BuO)4Ti, 93.1, 93.7, 4.6, 0.8, 0.5, 0.4. A similar run with (iso-PrO)3Al and II (98.5%-pure) gave a mixture of 91.5% II, 1.4% V, and 7.2% VI. I shaken with 10 or 5 mole-% aqueous acid at room temperature, neutralized with Na2CO3, washed with H2O, dried, and distilled gave an isomer mixture; a similar series of runs was performed with added Me2CO to achieve homogeneity; the mixture was kept 1 hr. at room temperature, neutralized with Na2CO3, and diluted with H2O to precipitate the isomer
mixture In this manner were prepared the following runs (aqueous catalyst and mole-% used, purity of I used, % content of I, IV, V, VI, and II in the resulting isomer mixture, and % polymer obtained are given): 40% HF, 10, 96.3, 94.7, 3.7, 0.8, 0.4, 0.4, 5; 44% HBF4, 10, 96.3, 93.6, 3.6, 0.8, 0.9, 1.7, --; 70% HClO4, 5, 96.3, 94.8, 3.5, 0.8, 0.5, 0.5, --; 40% HF (and Me2CO), 10, 97.1, 97.4, 1.1, 0.4, 0.5, 0.7, 2; 44% HBF4 (and Me2CO), 10, 97.1, 97.4, 0.9, 0.4, 0.4, 0.8, 2; 70% HClO4 (and Me2CO), 5, 97.1, 96.6, 0.9, 0.4, 0.7, 1.4, 5. I (97.1%-pure) (3.0 g.), 0.17 cc. 70% HClO4, and 10 cc. AcOH heated 5 min. at 100°, diluted with H2O, and worked up in the usual manner yielded a mixture of 93.1 I, 4.8 (IV), 0.9 V, 0.6 VI, 0.7% II, and some undecyl acetate. I (96.3%-pure) (3.0 g.) and 10 mole-%

concentrated H2SO4 stirred at -40° during 2 hrs., neutralized with excess aqueous Na2CO3, diluted with MeOH to break the emulsion, and extracted with hexane gave a mixture containing 86.3 I, 9.8 IV, 1.8 V, 1.0 VI, and 1.7% II, and 5% polymer. I (96.3%-pure) and 5 mole-% p-MeC6H4SO3H refluxed 5 hrs. gave 20% isomer mixture and 80% black oil and charred solid as residue; the isomer mixture consisted of 15.7 I, 28.3 IV, 31.9 V, 18.4 VI, and 5.7% II. I (96.3%-pure) (3.0 g.) and 5 mole-% 97-8% PhSO3H refluxed 1 hr. and distilled gave 80% mixture of 1.8 I, 28.5 IV, 28.2 V, 27.7 VI, and 13.9% II, and 20% oily polymer. I (96.3%-pure) (3.0 g.) and 10 mole-% C8H17Cl refluxed 10 hrs. (HCl evolved) and distilled yielded 50% mixture of 5.4 I, 34.4 IV, 28.3 V, 20.0 VI, and 11.8% II, and 50% oily residue. I (95.3%-pure) (3.0 g.) and 0.29 g. C8H17Cl refluxed 10 hrs. and ozonized without the previous removal of the C8H17Cl showed the presence of a mixture of 94.5 I, 3.2 IV, 1.1 V, 0.5 VI, and 0.7% II. I (97.1%-pure) (3.0 g.), 0.29 g. 70% HClO4, and a sufficient amount of Ac2O to remove the H2O kept 0.5 hr. at 100° gave a mixture of 4.0 I, 40.0 IV, 28.1 V, 18.4 VI, and 9.5% II and left 5% oily residue. I (96.3%-pure) (3.0 g.) and 0.201 g. AgClO4 refluxed 1 hr. yielded a mixture of 1.5 I, 26.9 IV, 26.7 V, 27.6 VI, and 17.3% II and left 5% oil . I (96.3%-pure) (3.0 g.) and 0.13 g. AlCl3 refluxed 5 hrs. and decomposed with excess 20% aqueous NaOH, and the organic layer worked up gave only polymer products. A similar run during 0.5 hr. yielded 50% mixture of 92.9 I, 4.6 IV, 1.2 V, 0.7 VI, and 0.7% II and 50% polymer. A similar run during 0.5 hr. in MeNO2 as solvent gave from 3.0 g. 97.1%-pure I a mixture of 78.4 I, 9.4 IV, 5.1 V, 4.1 VI, and 2.9% II and 20% polymer. I (96.3%-pure) (2.0 g.) treated slowly at room temperature with BF3 during 1.5 hrs. gave only polymer. A similar run during 5 min. gave a mixture of 82.3 I, 11.8 IV, 2.6 V, 1.4 VI, and 1.9% II, and 30% polymer. I (96.3%-pure) (3.0 g.) and 0.125 cc. Et2O.BF3 heated 5 hrs. at 120° gave a mixture of 56.7 I, 22.0 IV, 9.9 V, 7.1 VI, and 4.3 II and left 15% oily polymer. I (97.1%-pure) (3.0 g.), 0.125 cc. Et2O.BF3, and 3.0 cc. dry Et2O refluxed 3 hrs. gave a mixture of 97.3 I, 0.8 IV, 0.5 V, 0.6 VI, and 0.8% II and left 2% polymer. I (97.1%-pure) (3.0 g.) and 5 mole-% BF3.2AcOH, b15 64-6°, shaken 1 hr. at 25°, washed with aqueous Na2CO3 and H2O, dried, and distilled yielded a mixture M 96.1 I, 2.2 IV, 0.7 V, 0.5 VI, and 0.5% II and left 5% polymer. ofg (1.0 g.) in 50 cc. dry Et2O treated with 6.4 g. Br, the resulting 2-phase mixture treated with 6.0 g. 93.1%-pure I, kept 48 hrs. and distilled gave a mixture of 92.2 I, 5.6 IV, 1.2 V, 0.6 VI, and 0.5% II. MgBr2.3Et2O (0.04 mole) treated with dioxane, the crystalline dioxane-0.5 MgBr2 dissolved in 50 cc. refluxing PrOH, the solution refluxed 5 hrs. with 6 g. 93.1%-pure I, concentrated to remove the PrOH, filtered, and distilled gave a mixture of 91.8 I, 5.1 IV, 2.5 V, 0.3 VI, and 0.3% VII. MeI (1.42 g.) in Et2O converted with Mg to MeMgI, the Et2O replaced by 14 cc. dry iso-Am2O, a sufficient volume to make 20 mole-% MeI2 + MeMgI added to 6 g. 97.1%-pure I, the mixture heated 5 hrs. under Ar at 120°, decomposed with H2O, and worked up gave a mixture containing 88.4 I, 7.2 IV, 2.0 V, 1.0 VI, and 1.4% II, and left 8% polymer. A series of isomerization runs was performed with 3.0-g. amts. of I (of various purities) and 0.19 g. III at various temps. (in the runs at 190°, the undecene was distilled oil; the runs at lower temps. were ozonized without the previous removal of the III); at 190° the III decomposed with the formation of an Fe mirror, at 100° with the precipitation of a red-brown solid; the runs with simultaneous ultraviolet irradiation were performed in glass equipment under N or Ar. The following runs were performed in this manner (% purity of the I used, reaction temperature, reaction time in hrs., and % content of I, IV, V, VI, and II in the resulting undecene mixture given): 96.3, 20°, 65, 92.0,

5.2, 1.5, 0.8, 0.5; 96.3, 100°, 1, 70.9, 16.4, 6.8, 3.8, 2.1; 93.1, 190°, 0.17, 1.9, 29.9, 26.3, 27.1, 14.9; 93.1, 190, 0.17, 3.1, 25.8, 24.6, 26.4, 20.1; 93.1, 190° (irradiated before heating), 0.17, 2.1, 28.6, 25.0, 26.7, 17.5; 96.3, 190° (irradiated before heating), 0.17, 2.9, 28.6, 25.8, 28.0, 14.7. Similar runs were performed with II (same data given): 98.5, 190°, 0.17, 2.7, 14.3, 13.1, 16.8, 53.2; 98.5, 190°, 0.25, 3.2, 20.7, 19.6, 21.1, 35.5; 98.5, 190°, 0.5, 2.2, 20.2, 18.1, 20.8, 38.7; 98.5 (with 3-fold amount III), 190°, 3 times 0.25, 1.8, 25.9, 22.5, 24.2, 25.6. A series of runs with I was performed under simultaneous irradiation (same data given): 96.3, 50° (360-w. lamp), 5, 50.5, 20.3, 14.2, 9.5, 5.5; 97.1, 25° (360-w. lamp), 5, 70.7, 13.3, 8.2, 5.0, 2.8; 96.3, 25° (460-w. lamp), 5, 8.2, 35.4, 27.1, 19.5, 9.8. III (0.38 g.) shaken several hrs. under Ar at room temperature with 40 cc. 10% aqueous NaOH, the
solution treated with 3 g. 97.1%-pure I and acidified dropwise at 0° under Ar with 40 cc. cold 15% H2SO4, the mixture warmed during 1 hr. to 20°, kept 3 hrs. at 20°, and the organic phase worked up gave a mixture of 81.3 I, 12.5 IV, 2.9 V, 2.0 VI, and 1.3% II. III (2.8 cc.) in 12 cc. MeOH shaken under Ar a few min. with 6 cc. 50% aqueous NaOH, the mixture treated with 3.0 g. 97.1%-pure I in 12 cc. MeOH and 24 cc. iso-PrOH, kept 24 hrs. at 15°, and diluted with 60 cc. H2O precipitated a mixture of 58.2 I, 36.4 IV, 2.8 V, 1.5 VI, and 1.1 II. I (96.1%-pure) irradiated 0.5 and 5 hrs. with a 360-w. ultraviolet lamp gave a mixture of 91.7 I, 5.3 IV, 2.7 V, 0.4% II with a trace of II, and a mixture of 92.9 I, 4.9 IV, 1.1 V, 0.6 VI, and 0.5% II, resp. Bz2O2 (93%-pure) dissolved with 25% H2O in CHCl3 and repptd. with MeOH and dried, a 0.246-g. portion dissolved at 50-70° in 3 g. 96.3%-pure I, and the mixture heated 5 hrs. at 190° gave a mixture of 94.9 I, 3.6 IV, 0.6 V, 0.5 VI, and 0.4% II. Ph3CCl (1.4 g.) in 60 cc. dry C6H6 shaken 5 min. with 3.5 g. Zn dust, treated with 3.0 g. 96.3%-pure I, refluxed 5 hrs., shaken with air to convert the radical to the peroxide, concentrated, filtered, and distilled gave a mixture of 93.3 I, 4.7 IV, 1.1 V, 0.5VI, and 0.5% II.

L4 ANSWER 2 OF 3 CAPLUS

ACCESSION NUMBER:	1906:163714 CAPLUS
DOCUMENT NUMBER:	0:163714
TITLE:	On oxidations by means of ozone. [machine translation]
AUTHOR(S):	Harries, C.
CORPORATE SOURCE:	I. Chem. Inst. d. Univ., Berliu
SOURCE:	Berichte der Deutschen Chemischen Gesellschaft (1903), 36, 1933-36 From: Chem. Zentr., 1903, II, 188-189 CODEN: BDCGAS
DOCUMENT TYPE:	Journal
LANGUAGE:	Unavailable

AB [Machine Translation of Descriptors]. In the technology for a long time, ozone in larger scales are used than oxidants, for example, for the preparation of soluble strength and for the production of vanillin from isoeugenols, one of this medium was already rarely availed for scientific purposes. The principal reason for this lies probably in the fact that the SIEMENS and BERTHELOT tubes existing in the laboratories are not sufficient for the preparation of larger quantities of ozone. The author used the subsequent experiments with the larger, by SIEMENS & HALSKE apparatus which can be referred, in which the oxygen with currents of 5000 to 6000 volts voltage was ozonized. In such a way, gas mixture produced was proved as excellent medium for the preparation of aldehydes from unsaturated compounds. One leads strongly ozonized oxygen

into very well cooled Mesityl oxide, (CH3) 2C: CH·CO·CH3, then a thick, stinging smelling, yellow oil forms, which when taken out of the refrigerant, heats mixture with lower rattling noise automatically and then lower fire feature vehemently exploded. Certainly peroxide could be present here, (see original document for formula). Completely smooth and safe, the oxidation of the Mesityl oxide was carried out against it with the presence of water, lower formation of acetone, (CH3)2CO, and methyl glyoxal, CH3·CO·CHO. Analogous produces Methyl heptenone, (CH3)2C : CH·CH2·CH2·CO·CH3, acetone and Laevulin aldehyde (Pentanonal),CH3·CO·CH2·CH2·CHO. By the author, (Ber. German Chemistry Society vo. 31, pg. 37; C. vo. 98, I. pg. 448), in early times by cleavage of the Sylvane (α -Methyl furfuran) from the beech tar obtained Ketonaldehyde by means of ozonized oxygen, also represented completely smoothly from allyl acetone, CH2:CH·CH2·CH2· CO·CH3. The Methylphenyldihydropyridazine, (see original document for formula), available from it by condensation with phenylhydrazine diluted acid, melting, against the earlier indication, at 196 to 197° without decomposition. Analogous of these results would have the citral if the TIEMANN formula (CH3) 2C:CH·CH2·CH2·C(CH3): CH·CHO concerning the position of the double bonds is correct, with the oxidation with ozone Pentanonal to likewise be produced; in reality, however this aldehyde developed only in very small quantity, a very decomposable dialdehyde or Ketoaldehyde was the major product of the reaction. Also unsaturated aldehydes and acetals was easily contested by ozone. 3g Acrolein acetal, CH2:CH·CH(OC2H5)2, was oxidized for 1 1/2 hours to the Glyoxal semiacetal,CHO·CH(OC2H5)2,; stinging odor liquid; Kp760, 80 to 90° under decomposition, (apparent decay in alcohol and glyoxal); with Phenylhydrazinacetate, produces an oily hydrazone, that when heated, turns to the gold-yellow needles of the Glyoxalphenylosazone, solidifies to C6H5·NH·N : CH·CH:N·NH·C6H5. So far, oleic acid, cinnamic acid, maleic acid, and the Fumaric acid dimethyl ester were oxidized by unsaturated acid with ozone. The results with acid are not yet completely clear, the cinnamic acid slowly turned into benzaldehyde and glyoxylic acid, CHO·COOH. the preparation of latter acid could be more recommendable than the easily soluble maleic acid. When Fumaric acid ester suspends 2g, in 20 ccm water, were treated with ozone for about 2 hours, remained 1.4g of esters unmodified, during from the filtrate by Phenylhydrazinacetate 1g Glyoxylic acid methyl ester phenyl hydrazone, CH3OOC·CH:N·NH·C6H5, was precipitated. Gold-yellow needles from diluted methyl alcohol with melting point of 139°. Also alcohols are oxidized by ozone. Methyl alcohol in 50% solution produced formaldehyde; Glycerol in a solution of Phenylhydrazinacetate, produced the known osazone CH2 (OH) ·C(: N·NH·C6H5)·CH : N·NH·C6H5, the glyceraldehyde, CH2(OH)·CH(OH)·CHO. Dioxyacetone, CO(CH2·OH)2, was produced. Stilbene, C6H3·CH : CH·C6H5, was cleaved with the presence of water slowly, but smoothly to benzaldehyde. The oxidation with ozone, which would be allowed to make the easier production for a number was possible so far with difficulty accessible dialdehydes, Ketoaldehyde and Aldehyde acid, it is to be implemented always with caution, since some compounds let most explosive peroxides develop as intermediates. It is strange that these oxidations remain always standing with the formation of aldehydes: in no case could be isolated appropriate acid also are only in traces.

L4 ANSWER 3 OF 3 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 1906:158434 CAPLUS
DOCUMENT NUMBER: 0:158434
TITLE: On the ozonide of oleic acid. [machine translation]
AUTHOR(S): Harries, C.; Thieme, Carl
CORPORATE SOURCE: Kiel. Chem. Inst. d. Univ.
SOURCE: Berichte der Deutschen Chemischen Gesellschaft (1906), 39, 2844-46
From: Chem. Zentr., 1906, II, 1394
CODEN: BDCGAS
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

AB [Machine Translation of Descriptors]. Like the authors (LIEBIGs Ann. 343. 318; C. 1906. I. 542) showed, with the effect of O3 to a solution from oleic acid in chloroform, a syrupy compound is received, which results now from accumulation of 4 atoms O to the elements of oleic acid and Oil acid ozonide peroxide (I) which was mentioned. Peroxide forms a water-clear, nearly glassy mass for cases of its acetic ester solution with petroleum ether after that, deflagrates on that platinum sheet only weakly and decomposing with little water gradually under formation of azelaic acid. If one washes peroxide to syrupy with water and NaHCO3 and if the product was isolated with dried ether, then it changes under cleavage of an O-atom as H2O2 into normal oil acid ozonide (II), which is more highly liquid than peroxide. One can produce the normal ozonide directly from oleic acid by ozonized of its glacial acetic acid solution, washing with water and neutralization with bicarbonate. A colorless, high-viscosity oil , that is weakly deflagrates and decomposes with little water under separation of azelaic acid on the platinum sheet. The yield to normal ozonide is intermittent, since it does not succeed always, when washing peroxide with water the large cleavage in azelaic acid and Nonyl aldehyde and/or to avoid Nonylic acid. Solubility conditions of both ozonides are similar; the normal ozonide was not precipitated meanwhile from its acetic acid solution by petroleum ether. The two ozonides differ by their behavior when boiled with water , as peroxide gives a many more intensive H2O2-reaction than the normal ozonide. The earlier indication that peroxide indicates only with difficulty the H2O2-reaction, is to correct from this. The split products with water are qualitatively the same with both compounds: Azelainic acid half aldehyde, and/or Azelaic acid and Nonyl aldehyde, and/or Nonylic acid. The authors point the priority utterances, which MOLINARI and SONCINI (Annuario della society Chim. di Milano. Volume. XI. fasc. 12) reset, as unauthorized.

=> d his

(FILE 'HOME' ENTERED AT 16:51:32 ON 16 JAN 2008)

FILE 'CAPLUS' ENTERED AT 16:51:59 ON 16 JAN 2008

```
L1          9 S ORGANIC (L) OZONIDES (L) (MAKE OR PRODUCE)
L2        203 S OZONIZED (L) (FAT? OR OIL?) (L) WATER
L3          0 S L2 AND (PRODUCE OR MAKE) (L) PEROXICE
L4          3 S L2 AND (PRODUCE OR MAKE) (L) PEROXIDE
```

=> s (oil ajd water) (L) (ozone or oxygen) and peroxide

```
        807480 OIL
        386515 OILS
        912810 OIL
                 (OIL OR OILS)
             4 AJD
       2650614 WATER
        271094 WATERS
       2708560 WATER
                 (WATER OR WATERS)
             0 OIL AJD WATER
                 (OIL(W)AJD(W)WATER)
         87996 OZONE
            47 OZONES
         88004 OZONE
                 (OZONE OR OZONES)
        806169 OXYGEN
          7275 OXYGENS
        811237 OXYGEN
                 (OXYGEN OR OXYGENS)
             0 (OIL AJD WATER) (L) (OZONE OR OXYGEN)
        225166 PEROXIDE
         48785 PEROXIDES
        244405 PEROXIDE
                 (PEROXIDE OR PEROXIDES)
L5           0 (OIL AJD WATER) (L) (OZONE OR OXYGEN) AND PEROXIDE

=> s (oil (4w) water) (L) (ozone and oxygen) with peroxide
MISSING OPERATOR OXYGEN) WITH
The search profile that was entered contains terms or
nested terms that are not separated by a logical operator.

=> s (oil (4w) water) (L) (ozone and oxygen) (L) peroxide
PROXIMITY OPERATION NOT ALLOWED
Certain operators may not be nested in combination with other
operators.  A nested operator is valid only when it occurs at the same
level or above the operator outside the nested phrase as determined by
the following precedence list:

                1.    Numeric
                2.    (W), (NOTW), (A), (NOTA)
                3.    (S), (NOTS)
                4.    (P), (NOTP)
                5.    (L), (NOTL)
                6.    AND, NOT
                7.    OR

For example, '(MONOCLONAL(W)ANTIBOD?)(L)ANTIGEN?' is valid since (W)
is above (L) on the precedence list.  However,
'((THIN(W)LAYER)(L)PHOSPHOLIPID#)(A)LACTONE#' is not valid since (L)
is below (A) on the precedence list.  The only exception is the 'OR'
operator.  This operator may be used in combination with any other
operator.  For example, '(ATOMIC OR NUCLEAR)(W)REACTOR' is valid.

=> s(oil (4w) water) (s) (ozone or oxygen) (W) peroxide
S(OIL IS NOT A RECOGNIZED COMMAND
The previous command name entered was not recognized by the system.
For a list of commands available to you in the current file, enter
"HELP COMMANDS" at an arrow prompt (=>).
```

```
=> s (oil (4w) water) (S) (oxygen or ozone)
        807480 OIL
        386515 OILS
        912810 OIL
                  (OIL OR OILS)
       2650614 WATER
        271094 WATERS
       2708560 WATER
                  (WATER OR WATERS)
        806169 OXYGEN
          7275 OXYGENS
        811237 OXYGEN
                  (OXYGEN OR OXYGENS)
         87996 OZONE
            47 OZONES
         88004 OZONE
                  (OZONE OR OZONES)
L6         268 (OIL (4W) WATER) (S) (OXYGEN OR OZONE)

=> s l6 and peroxide
        225166 PEROXIDE
         48785 PEROXIDES
        244405 PEROXIDE
                  (PEROXIDE OR PEROXIDES)
L7          16 L6 AND PEROXIDE

=> d his

     (FILE 'HOME' ENTERED AT 16:51:32 ON 16 JAN 2008)

     FILE 'CAPLUS' ENTERED AT 16:51:59 ON 16 JAN 2008
L1              9 S ORGANIC (L) OZONIDES (L) (MAKE OR PRODUCE)
L2            203 S OZONIZED (L) (FAT? OR OIL?) (L) WATER
L3              0 S L2 AND (PRODUCE OR MAKE) (L) PEROXICE
L4              3 S L2 AND (PRODUCE OR MAKE) (L) PEROXIDE
L5              0 S (OIL AJD WATER) (L) (OZONE OR OXYGEN) AND PEROXIDE
L6            268 S (OIL (4W) WATER) (S) (OXYGEN OR OZONE)
L7             16 S L6 AND PEROXIDE

=> s l7 not l4 not l1
L8          16 L7 NOT L4 NOT L1

=> s l8 and (process or make)
       2551624 PROCESS
       1736702 PROCESSES
       3802963 PROCESS
                  (PROCESS OR PROCESSES)
        279272 MAKE
        216488 MAKES
        480012 MAKE
                  (MAKE OR MAKES)
L9           7 L8 AND (PROCESS OR MAKE)

=> d l9 1-7 ibib abs

L9   ANSWER 1 OF 7  CAPLUS  COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER:        2007:1377201  CAPLUS
```

DOCUMENT NUMBER: 148:56934
TITLE: Preparation of azelaic acid with solid acid catalyst
INVENTOR(S): Chen, Yepu; Shi, Chunwei; Chen, Xin
PATENT ASSIGNEE(S): Shanghai University, Peop. Rep. China
SOURCE: Faming Zhuanli Shenqing Gongkai Shuomingshu, 8pp.
CODEN: CNXXEV
DOCUMENT TYPE: Patent
LANGUAGE: Chinese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
CN 101077856	A	20071128	CN 2007-10042766	20070626

PRIORITY APPLN. INFO.: CN 2007-10042766 20070626

AB Title method comprises mixing unsatd. acid and fatty acid, mixed fatty acid from animal and plant oil (hydrolysis), solid acid catalyst, hexadecyl tri-Me ammonium chloride, heating to 50-70 °C, leading in gas mixture with ozone concentration of 2-10 g/L, adding 20-40% hydrogen peroxide, reacting at 95-110 °C for 5-12 h, separating solid acid catalyst, oil phase and water phase; crystallization of water phase at 3 °C for white azelaic acid, filtering, drying; boiling water extracting oil phase, crystallization at 3°C, vacuum filtering for azelaic acid; combining azelaic acid crystal, recrystn. in hot water for pure azelaic acid. The solid acid catalyst is SO42-/ZrO2-Al2O3, SO42-/TiO2, or phosphotungstic acid loaded 5A type macroporous mol. sieve.

L9 ANSWER 2 OF 7 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 2006:1257685 CAPLUS
DOCUMENT NUMBER: 146:486391
TITLE: Oxidative degradation of olive oil mill waste water
AUTHOR(S): Bentivenga, G.; D'auria, M.; Emanuele, L.; Racioppi, R.
CORPORATE SOURCE: Corleto Perticara, ITIS A. Einstein, Potenza, Italy
SOURCE: Rivista Italiana delle Sostanze Grasse (2006), 83(4), 158-161
CODEN: RISGAD; ISSN: 0035-6808
PUBLISHER: S.E.A. - Servizi Editoriali Associati srl
DOCUMENT TYPE: Journal
LANGUAGE: English

AB The treatment of diluted olive oil mill waste water with Fenton's reagent reduces COD. The reaction followed a zero order kinetics. The reaction needs to use large amts. of reagents to have an appreciable reduction of COD. Treatment of olive oil mill waste water with ozone reduces COD. The reaction followed a first order kinetics. The UV spectrum of olive oil mill waste water after treatment with ozone did not show absorptions.

REFERENCE COUNT: 26 THERE ARE 26 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L9 ANSWER 3 OF 7 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 2006:199913 CAPLUS
DOCUMENT NUMBER: 144:356419
TITLE: Advanced treatment of oil recovery wastewater from polymer flooding by UV/H2O2/O3 and fine filtration
AUTHOR(S): Ren, Guang-meng; Sun, De-zhi; Chunk, Jong Shik
CORPORATE SOURCE: School of Municipal and Environmental Engineering,

Harbin Institute of Technology, Harbin, 150090, Peop. Rep. China

SOURCE: Journal of Environmental Sciences (Beijing, China) (2006), 18(1), 29-32
CODEN: JENSEE; ISSN: 1001-0742

PUBLISHER: Science Press

DOCUMENT TYPE: Journal

LANGUAGE: English

AB In order to purify oil recovery wastewater from polymer flooding (ORWPF) in tertiary oil recovery in oil fields, advanced treatment of UV/H2O2/O3 and fine filtration were investigated. The exptl. results showed that polyacrylamide and oil remaining in ORWPF after the conventional treatment process could be effectively removed by UV/H2O2/O3 process
. Fine filtration gave a high performance in eliminating suspended solids. The treated ORWPF can meet the quality requirement of the wastewater-bearing polymer injection in oilfield and be safely re-injected into oil reservoirs for oil recovery.

REFERENCE COUNT: 17 THERE ARE 17 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L9 ANSWER 4 OF 7 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 2000:827415 CAPLUS

DOCUMENT NUMBER: 134:151896

TITLE: Photocatalytic/H2O2 treatment of oil field produced waters

AUTHOR(S): Bessa, E.; Sant'Anna, G. L.; Dezotti, M.

CORPORATE SOURCE: Centro de Tecnologia, COPPE, Programa de Engenharia Quimica (PEQ), Water Pollution Control Lab, Universidade Federal do Rio de Janeiro (UFRJ), Rio de Janeiro, RJ, 21945-970, Brazil

SOURCE: Applied Catalysis, B: Environmental (2001), 29(2), 125-134
CODEN: ACBEE3; ISSN: 0926-3373

PUBLISHER: Elsevier Science B.V.

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Gas chromatog.-mass spectroscopy (GC-MS) evaluated the efficiency of photocatalytic degradation of pollutants in oil field produced water from Campos Basin, Rio de Janeiro, Brazil, over TiO2 (anatase), in terms of organic loading reduction and acute toxicity removal observed in a previous publication. The effect of adding H2O2, studied through an exptl. design, was adverse. Two types of photocatalyst (Aldrich and Degussa P25) were used; afterward, their surfaces were examined using SEM. The former showed an intense initial coverage by organic compds. and a severe corrosion with the use of H2O2; the latter presented almost no initial coverage and was not corroded for small periods of photocatalytic treatment, also in the presence of H2O2.

REFERENCE COUNT: 26 THERE ARE 26 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L9 ANSWER 5 OF 7 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1999:238946 CAPLUS

DOCUMENT NUMBER: 130:242069

TITLE: Process of treating produced water with ozone

INVENTOR(S): Morrow, Lawrence Robert; Martir, Wilson Kirkpatrick; Wright, David Edmund; Aghazeynali, Hossein

PATENT ASSIGNEE(S): Texaco Development Corporation, USA

SOURCE: Can. Pat. Appl., 40 pp.
CODEN: CPXXEB
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.		DATE
CA 2203201	A1	19980228	CA 1997-2203201		19970421
US 5868945	A	19990209	US 1996-741657		19960829
PRIORITY APPLN. INFO.:			US 1996-741657	A	19960829

AB Ozonolysis is an effective process for improving the quality of produced water. A process for reducing the concentration of water soluble organic materials dissolved in produced water consists of introducing into the produced water a sufficient amount of gaseous ozone by use of a means for maximizing the collision frequency of ozone gas and the produced water. The temperature of the produced water is between from about 80° to about 180° F. The process renders a water effluent with markedly reduced oil and grease content. The water effluent can be used on land as a drinking or irrigation water supply source and may be safely discharged into navigable waters.

L9 ANSWER 6 OF 7 CAPLUS
ACCESSION NUMBER: 1997:248039 CAPLUS
DOCUMENT NUMBER: 126:313799
TITLE: Electrochemical Generation and Reactions of Ferrylmyoglobins in Water and Microemulsions
AUTHOR(S): Onuoha, Anthony C.; Zu, Xiaolin; Rusling, James F.
CORPORATE SOURCE: Department of Chemistry, University of Connecticut, Storrs, CT, 06269-4060, USA
SOURCE: Journal of the American Chemical Society (1997), 119(17), 3979-3986
CODEN: JACSAT; ISSN: 0002-7863
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
AB Ferrylmyoglobin species, which are active oxidant forms of the protein myoglobin, were obtained by electrochem. reduction of metmyoglobin [MbFeIII] in the presence of oxygen in aqueous neutral buffer and in microemulsions of oil, water, and cationic surfactant. Reduction of myoglobin at -0.4 V vs. SCE catalyzed the reduction of oxygen to hydrogen peroxide at the electrode. Hydrogen peroxide oxidizes metmyoglobin in solution to give the radical ferrylmyoglobin •X-MbFeIV=O, which is known to decay rapidly to the non-radical MbFeIV=O. This complex reduction-oxidation process converted nearly all of the 30 μM myoglobin in a spectroelectrochem. cell to ferrylmyoglobins in 15 min in pH 7.3 buffer and in 18 min in microemulsions. Characteristic ferryl heme absorbance bands near 421, 548, and 584 nm were used to identify products. Confirmation of ferrylmyoglobins was provided by redns. to metmyoglobin with ascorbate and by myoglobin-mediated electrochem. epoxidn. of styrene. Fiftyfold higher yields of styrene oxide and benzaldehyde were achieved in a microemulsion compared to electrochem. or chemical methods in pH 7.4 buffer. The electrochem. approach described may also prove useful for investigating active catalytic species of heme enzymes such as cytochrome P 450.
REFERENCE COUNT: 59 THERE ARE 59 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

```
L9   ANSWER 7 OF 7  CAPLUS  COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER:         1989:557274  CAPLUS
DOCUMENT NUMBER:          111:157274
TITLE:                    Process for converting sulfide ion to
                          innocuous, solubile sulfur species
INVENTOR(S):              Allison, Joe D.; Thomas, Sally A.; Yang, Kang
PATENT ASSIGNEE(S):       Conoco, Inc., USA
SOURCE:                   U.S., 13 pp. Cont. of U.S. Ser. No. 947,442,
                          abandoned.
                          CODEN: USXXAM
DOCUMENT TYPE:            Patent
LANGUAGE:                 English
FAMILY ACC. NUM. COUNT:   1
PATENT INFORMATION:

     PATENT NO.          KIND    DATE        APPLICATION NO.         DATE
     ---------------     ----    --------    --------------------    --------
     US 4839154           A      19890613    US 1988-199721          19880531
PRIORITY APPLN. INFO.:                       US 1986-947442       A1 19861229
AB   The title process includes contacting the sulfide ion in
     oil-field,produced waters with an oxidizing agent to oxidize it to the
     water-soluble S species and mixing a surfactant with the solution in an amount
     sufficient to inhibit precipitation of elemental S and to promote subsequent
     oxidation  To enhance the formation of the water-soluble S species and to speed
     up the reaction, the oxidation is conducted under alkaline conditions.  The
     process increases water flooding capabilities and makes
     disposal of the emerging waters easier to handle and environmentally safe.


=> d his

     (FILE 'HOME' ENTERED AT 16:51:32 ON 16 JAN 2008)

     FILE 'CAPLUS' ENTERED AT 16:51:59 ON 16 JAN 2008
L1              9 S ORGANIC (L) OZONIDES (L) (MAKE OR PRODUCE)
L2            203 S OZONIZED (L) (FAT? OR OIL?) (L) WATER
L3              0 S L2 AND (PRODUCE OR MAKE) (L) PEROXICE
L4              3 S L2 AND (PRODUCE OR MAKE) (L) PEROXIDE
L5              0 S (OIL AJD WATER) (L) (OZONE OR OXYGEN) AND PEROXIDE
L6            268 S (OIL (4W) WATER) (S) (OXYGEN OR OZONE)
L7             16 S L6 AND PEROXIDE
L8             16 S L7 NOT L4 NOT L1
L9              7 S L8 AND (PROCESS OR MAKE)

=> s (fat# or oil#) (S) water (s) (ozone or oxygen)
        309772 FAT#
        932617 OIL#
       2650614 WATER
        271094 WATERS
       2708560 WATER
                 (WATER OR WATERS)
         87996 OZONE
            47 OZONES
         88004 OZONE
                 (OZONE OR OZONES)
        806169 OXYGEN
          7275 OXYGENS
```

```
        811237 OXYGEN
                 (OXYGEN OR OXYGENS)
L10         852 (FAT# OR OIL#) (S) WATER (S) (OZONE OR OXYGEN)

=> l10 peroxide (5w) product
L10 IS NOT A RECOGNIZED COMMAND
The previous command name entered was not recognized by the system.
For a list of commands available to you in the current file, enter
"HELP COMMANDS" at an arrow prompt (=>).

=> s l10 peroxide (5w) product
MISSING OPERATOR L10 PEROXIDE
The search profile that was entered contains terms or
nested terms that are not separated by a logical operator.

=> s l10 and (peroxide (5w) product)
        225166 PEROXIDE
         48785 PEROXIDES
        244405 PEROXIDE
                 (PEROXIDE OR PEROXIDES)
       1253455 PRODUCT
       1473226 PRODUCTS
       2376779 PRODUCT
                 (PRODUCT OR PRODUCTS)
          2969 PEROXIDE (5W) PRODUCT
L11           2 L10 AND (PEROXIDE (5W) PRODUCT)

=> d l11 1-2 ibib abs
```

L11 ANSWER 1 OF 2 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 1963:403034 CAPLUS
DOCUMENT NUMBER: 59:3034
ORIGINAL REFERENCE NO.: 59:448g-h,449a
TITLE: Organic peroxides and synthetic peroxidases
PATENT ASSIGNEE(S): Olea-Centre
SOURCE: 15 pp.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
BE 617329		19620831	BE	
FR 1322134			FR	
GB 1012664			GB	

PRIORITY APPLN. INFO.: FR 19610630

AB Stable organic peroxides which are water-soluble are prepared by treating with ozone a dilute acid solution of a mixed ether of polyethylene glycol and castor oil, named ERP. The best yield is obtained with an ERP concentration of 1% and a temperature of 60°. The peroxides obtained are epoxy compds. Two complex compds. are prepared by treating the peroxide solution with copper acetate (I) or iodine; alternatively, the complex compds. may be prepared by treating the ERP solution with ozone in the presence of I or iodine. The solution of the copper peroxide compound (ERP-Cu) acts as a synthetic peroxidase, inhibiting at high concentration and at pH<7 the oxidizing properties of H2O2 or organic peroxides, and at low concentration, the product acts as a

catalyst. The solution of the iodine peroxide compound (ERP-iodine), ৷ does not color starch, acts in alkaline medium releasing slowly iodine production of hypoiodous acid that is a powerful oxidizing agent. At t level, the ERP-Cu could be employed as a hydrogen peroxide carrier and ERP-iodine could inhibit cancerous cells.

L11 ANSWER 2 OF 2 CAPLUS

ACCESSION NUMBER: 1906:135588 CAPLUS
DOCUMENT NUMBER: 0:135588
TITLE: PRODUCTION OF DISINFECTANTS
INVENTOR(S): Kingzett, Charles T.; Zingler, Maxmilian
PATENT ASSIGNEE(S): UK
SOURCE: U.S.
CODEN: USXXAM
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 274789	A	18830327	US 1882-113904	18821212

AB To all whom it may concern: Be it known that we, CHARLES THOMAS KINGZETT, F. I. C., F. C. S., and MAXIMILIAN ZINGLER, F. C. S., subjects of the Queen, of Great Britain, residing, respectively, at 17 Lansdowne Road, Tottenham, and 19 Buckland Crescent, Belsize Park, both in the county of Middlesex, England, have invented certain new and useful Improvements in the Production of Antiseptics and Disinfectants, (for which we have received Letters Patent in Great Britain, Number 274, dated January 24, 1876; in France, Number 113,904, dated July 24, 1876; in Belgium, Number 40,103, dated July 24, 1876; in Italy, Number 83, dated August 9, 1878, and in Austria, Number 50, dated September 16, 1877); and we do hereby declare that the following is a full, clear, and exact description of the invention, which will enable others skilled in the art to which it appertains to make and use the same. Our invention consists in the production of improved antiseptics and disinfectants as follows: Turpentine or rosin-spirit, or any essential oil or hydrocarbon which contains a terpene or which contains cymene, we expose to the action of atmospheric oxygen or pure oxygen in the presence of water . The hydrocarbon suffers oxidation and yields peroxide of hydrogen as a product of such change. We oxidize the hydrocarbon by air or oxygen, either in the presence or absence of water. We prefer to employ turpentine as the substance to be oxidized and atmospheric air as the agent of oxidation.

=> d his

(FILE 'HOME' ENTERED AT 16:51:32 ON 16 JAN 2008)

FILE 'CAPLUS' ENTERED AT 16:51:59 ON 16 JAN 2008

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L1          9 S ORGANIC (L) OZONIDES (L) (MAKE OR PRODUCE)
L2        203 S OZONIZED (L) (FAT? OR OIL?) (L) WATER
L3          0 S L2 AND (PRODUCE OR MAKE) (L) PEROXICE
L4          3 S L2 AND (PRODUCE OR MAKE) (L) PEROXIDE
L5          0 S (OIL AJD WATER) (L) (OZONE OR OXYGEN) AND PEROXIDE
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L6          268 S (OIL (4W) WATER) (S) (OXYGEN OR OZONE)
L7           16 S L6 AND PEROXIDE
L8           16 S L7 NOT L4 NOT L1
L9            7 S L8 AND (PROCESS OR MAKE)
L10         852 S (FAT# OR OIL#) (S) WATER (S) (OZONE OR OXYGEN)
L11           2 S L10 AND (PEROXIDE (5W) PRODUCT)

=> s (sunflower adj oil)(s) water (S) (ozone or oxyten)
          27978 SUNFLOWER
           2385 SUNFLOWERS
          28396 SUNFLOWER
                  (SUNFLOWER OR SUNFLOWERS)
            283 ADJ
         807480 OIL
         386515 OILS
         912810 OIL
                  (OIL OR OILS)
              0 SUNFLOWER ADJ OIL
                  (SUNFLOWER(W)ADJ(W)OIL)
        2650614 WATER
         271094 WATERS
        2708560 WATER
                  (WATER OR WATERS)
          87996 OZONE
             47 OZONES
          88004 OZONE
                  (OZONE OR OZONES)
             22 OXYTEN
              1 OXYTENS
             23 OXYTEN
                  (OXYTEN OR OXYTENS)
L12           0 (SUNFLOWER ADJ OIL)(S) WATER (S) (OZONE OR OXYTEN)

=> s (vegetable (A) oil (S) (water) (S) (ozone or oxygen)
UNMATCHED LEFT PARENTHESIS '(VEGETABLE'
The number of right parentheses in a query must be equal to the
number of left parentheses.

=> s (vegetable (A) oil) (S) water (S) (ozone or oxygen)
          99579 VEGETABLE
          31802 VEGETABLES
         114732 VEGETABLE
                  (VEGETABLE OR VEGETABLES)
         807480 OIL
         386515 OILS
         912810 OIL
                  (OIL OR OILS)
        2650614 WATER
         271094 WATERS
        2708560 WATER
                  (WATER OR WATERS)
          87996 OZONE
             47 OZONES
          88004 OZONE
                  (OZONE OR OZONES)
         806169 OXYGEN
           7275 OXYGENS
         811237 OXYGEN
```

```
              (OXYGEN OR OXYGENS)
L13          11 (VEGETABLE (A) OIL) (S) WATER (S) (OZONE OR OXYGEN)

=> s l13 not l10
L14           0 L13 NOT L10

=> s l13 not l8
L15          10 L13 NOT L8

=> d l15 1-10 ibib abs

L15  ANSWER 1 OF 10  CAPLUS  COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER:         2007:1056843  CAPLUS
TITLE:                    Method for producing of canned food mixed salad with
                          squids
INVENTOR(S):              Kvasenkov, O. I.; Yushina, E. A.; Gracheva, A. Y.
PATENT ASSIGNEE(S):       Vserossiyskii Nauchno-Issledovatel'skii Institut
                          Konservnoi I Ovoshchesushil'noi Promyshlennosti
                          (Gosudarstvennoe Nauchnoe Uchrezhdenie), Russia
SOURCE:                   Russ.
                          CODEN: RUXXE7
DOCUMENT TYPE:            Patent
LANGUAGE:                 Russian
FAMILY ACC. NUM. COUNT:   1
PATENT INFORMATION:

     PATENT NO.          KIND    DATE          APPLICATION NO.         DATE
     ---------------     ----    --------      --------------------    --------
     RU 2305993           C1     20070920      RU 2006-108766          20060321
PRIORITY APPLN. INFO.:                         RU 2006-108766          20060321
AB   FIELD: production of canned snack foods.SUBSTANCE: method involves cutting
     and blanching squids and potato; blanching and cutting carrot and beet;
     shredding and freezing fresh cabbage; cutting salted cucumbers, bulb
     onion, green onion and greens; mixing said components, without
     oxygen access, with vegetable oil, acetic
     acid, sugar, edible salt and black bitter pepper; packing resultant
     mixture and sweet water; pressurizing and sterilizing.EFFECT:
     provision for producing of canned food having increased digestibility.

L15  ANSWER 2 OF 10  CAPLUS  COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER:         2007:502070  CAPLUS
DOCUMENT NUMBER:          146:499896
TITLE:                    Method for preparing candies from vegetables, melons,
                          fruits and coarse cereal
INVENTOR(S):              Hu, Junmin
PATENT ASSIGNEE(S):       Wuhan Hugong Natural Food Co., Ltd., Peop. Rep. China
SOURCE:                   Faming Zhuanli Shenqing Gongkai Shuomingshu, 9pp.
                          CODEN: CNXXEV
DOCUMENT TYPE:            Patent
LANGUAGE:                 Chinese
FAMILY ACC. NUM. COUNT:   1
PATENT INFORMATION:

     PATENT NO.          KIND    DATE          APPLICATION NO.         DATE
     ---------------     ----    --------      --------------------    --------
     CN 1954687           A      20070502      CN 2005-10019684        20051028
PRIORITY APPLN. INFO.:                         CN 2005-10019684        20051028
AB   The title method comprises the steps of: (1) picking fresh vegetables,
```

potherbs, and flowers, removing grass and yellow leaves, cleaning, sterilizing in 80-90C water for 3-5min, processing into vegetable juice and flower juice resp., concentrating, and sterilizing, (2) decorticating melons and fruits, removing seeds, cleaning, processing into juice, concentrating, and sterilizing, (3) removing ashes and sand of coarse cereal, cleaning, drying naturally, and processing into syrup, (4) mixing vegetable juice, flower juice and condensate of melons, fruits, corns, Castanea mollissima, Ipomoea batatas, and Apios americana at a ratio of 1:0.1:1, and adding coarse cereal syrup, (5) adding milk powder, vegetable oil, xylitol and lotus root starch at a ratio of 4.5:0.5:1:2, (6) stirring homogeneously, (7) concentrating, (8) molding, (9) vacuum-drying, (10) cutting, and sterilizing, (11) oven-drying, (12) drying naturally, packaging, aerating the package bag with mixed gases of oxygen and nitrogen, sealing, incasing, and transferring into a storage.

L15 ANSWER 3 OF 10 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 2006:969962 CAPLUS
TITLE: Method for producing of canned food mutton fry
INVENTOR(S): Kvasenkov, O. I.; Yushina, E. A.
PATENT ASSIGNEE(S): Kvasenkov Oleg Ivanovich, Russia
SOURCE: Russ.
CODEN: RUXXE7
DOCUMENT TYPE: Patent
LANGUAGE: Russian
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
RU 2283600	C1	20060920	RU 2005-105113	20050225
PRIORITY APPLN. INFO.:			RU 2005-105113	20050225

AB FIELD: food-processing industry, in particular, preparing of canned meat-and-vegetable foods.SUBSTANCE: method involves cutting mutton and larding with garlic; shredding and freezing fresh cabbage; cutting fat, green onion and greens; cooking rice; rubbing "Parmesan" cheese; mixing mentioned components, without access of oxygen, with vegetable oil, edible salt and CO2-extract of pyrolytic wood; preparing media by mixing dry red wine and sweet water; packing mixture with media; pressurizing and sterilizing.EFFECT: provision for producing of canned food having improved digestibility as compared to similar culinary dish.

L15 ANSWER 4 OF 10 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 2006:700377 CAPLUS
TITLE: Method for manufacturing canned food named caviar-vegetable baked pudding
INVENTOR(S): Kvasenkov, O. I.; Grigorenko, S. P.; Maksyuta, I. V.; Yushina, E. A.
PATENT ASSIGNEE(S): Russia
SOURCE: Russ.
CODEN: RUXXE7
DOCUMENT TYPE: Patent
LANGUAGE: Russian
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE

RU 2279824	C2	20060720	RU 2004-114336	20040512
PRIORITY APPLN. INFO.:			RU 2004-114336	20040512

AB FIELD: food industry, canned food industry.SUBSTANCE: the present innovation deals with manufacturing canned food out of caviar and vegetables. It is necessary to prepare formula components: chopping and freezing fresh white cabbage, cutting and stewing onions in vegetable oil, mixing at heating up to 65-70° C at oxygen-free supply of caviar, cabbage, wheat flour, vegetable oil, brown onions, acetic acid, bitter black pepper, food structurizing concentrate and water at certain expenditure of components. The mixture obtained should be packages, hermetically sealed and sterilized. This provides prolonged terms of storage and increases digestibility of the target product.EFFECT: higher efficiency of manufacturing.

L15 ANSWER 5 OF 10 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 2006:589209 CAPLUS
TITLE: Method for producing of canned food stuffed cabbage rolls in sour cream sauce with tomatoes
INVENTOR(S): Kvasenkov, O. I.; Yushina, E. A.
PATENT ASSIGNEE(S): Kvasenkov Oleg Ivanovich, Russia
SOURCE: Russ.
CODEN: RUXXE7
DOCUMENT TYPE: Patent
LANGUAGE: Russian
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
RU 2277802	C2	20060620	RU 2004-117052	20040607
PRIORITY APPLN. INFO.:			RU 2004-117052	20040607

AB FIELD: production of canned meat-and-vegetable food.SUBSTANCE: method involves using farce including frozen shredded cabbage, onion thermally treated in vegetable oil, parsley greens of parsley, dill and celery ground by means of grinder; mixing farce without oxygen access; forming farce in frozen and defrosted cabbage leaves for producing of rolls; preparing sauce with the use of bone soup and water; boiling it out until dry substance content is 25%; pressurizing and sterilizing packaged stuffed cabbage rolls with sauce.EFFECT: increased digestibility of base product.

L15 ANSWER 6 OF 10 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 2006:409186 CAPLUS
DOCUMENT NUMBER: 144:393159
TITLE: Mild production of anhydrous vegetable oil
INVENTOR(S): Zimmer, Bruno
PATENT ASSIGNEE(S): Germany
SOURCE: Ger. Offen., 2 pp.
CODEN: GWXXBX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE

DE 102004053528 A1 20060504 DE 2004-102004053528 20041103
PRIORITY APPLN. INFO.: DE 2004-102004053528 20041103

AB To avoid damages of vegetable oils by effect of light and oxygen during removal of water, the treatment of the vegetable oil is carried out under inert atmospheric (N) and exclusion of light. The oil is fed into a nitrogen filled storage container passing a flow heater, where it is warmed to >100° (especially 103-105°), then to an reduced pressure-container, where the gaseous water may be removed, and subsequently to a nitrogen filled storage container, where the product is cooled down.

L15 ANSWER 7 OF 10 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 2004:917951 CAPLUS
DOCUMENT NUMBER: 142:154339
TITLE: Lipopeptide production from Bacillus sp. GB16 using a novel oxygenation method
AUTHOR(S): Lee, Baek-Seok; Kim, Eun-Ki
CORPORATE SOURCE: Department of Biological Engineering, Inha University, Incheon, 402-751, S. Korea
SOURCE: Enzyme and Microbial Technology (2004), 35(6-7), 639-647
CODEN: EMTED2; ISSN: 0141-0229
PUBLISHER: Elsevier B.V.
DOCUMENT TYPE: Journal
LANGUAGE: English

AB During lipopeptide (biosurfactant) production by Bacillus species GB16, conventional aeration method using added antifoam agent was unsuccessful due to the excessive formation of foam and the inhibitory effects of the antifoam chemical A novel integrated method was developed to increase the dissolved oxygen concentration during the microbial production of biosurfactant lipopeptides. This novel method consisted of adding hydrogen peroxide to the medium, which decomposed to oxygen and water by cell's catalase and adding a vegetable oil and Ca-stearate to the medium as antifoam agents, as well as oxygen vectors. The dissolved oxygen concentration could be controlled by the automatic addition of hydrogen peroxide to the bioreactor. A significant improvement, i.e., suppression of foaming and, therefore, a three-fold extension of the cultivation time and, consequently, remarkable increase in the lipopeptide production could be achieved. This result showed that a novel aeration method was effective, especially when excessive foaming caused problems during microbial production of biosurfactant.

REFERENCE COUNT: 47 THERE ARE 47 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L15 ANSWER 8 OF 10 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 2000:542418 CAPLUS
DOCUMENT NUMBER: 133:166102
TITLE: Method and device for refining vegetable oil and waste oil with ozone to be used as fuel in Diesel engine
INVENTOR(S): Matsumura, Masatoshi; Murakami, Seishiro
PATENT ASSIGNEE(S): Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 19 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.		DATE
JP 2000219886	A	20000808	JP 1999-24464		19990201
EP 1026224	A1	20000809	EP 2000-101951		20000201
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO					
US 6364917	B1	20020402	US 2000-496097		20000201
PRIORITY APPLN. INFO.:			JP 1999-24464	A	19990201

AB The refining process is carried out by heating the vegetable oil (virgin oil) and waste vegetable oil (e.g., from food processing) to 40-80°, charging 3-8% heated ozone water (40-80°) into the oil with charging 4000-30,000 ppm ozone gas from the bottom under agitation for separating impurity (e.g., glycerin and fatty acid) and water from the oil for removal to obtain pre-treated oil, then heating the pre-treated oil in a treatment tank at 40-80° with charging 4000-30,000 ppm micro-particular ozone gas from the bottom under agitation for secondary treatment, to obtain refined oil suitable as fuel for Diesel engine with clean exhaust gas.

L15 ANSWER 9 OF 10 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 1995:574207 CAPLUS
DOCUMENT NUMBER: 123:8205
TITLE: Influence of sorbed vegetable oil and relative humidity on the oxygen transmission rate through various polymer packaging films
AUTHOR(S): Johansson, F.; Leufven, A.
CORPORATE SOURCE: SIK, Swedish Institute for Food Research, Goeteborg, S-402 29, Swed.
SOURCE: Packaging Technology & Science (1994), 7(6), 275-81
CODEN: PTSCEQ; ISSN: 0894-3214
DOCUMENT TYPE: Journal
LANGUAGE: English

AB In this study, the effect of a vegetable oil on the oxygen barrier properties of different polymer packaging materials [i.e. amorphous polyethylene terephthalate (APET), polypropylene (PP) and high d. polyethylene (HDPE)] was investigated. The influence of both relative humidity and the combination of relative humidity and sorbed oil on the oxygen transmission rate (OTR) was studied. Regardless of the relative humidity, the APET film remained an excellent oxygen barrier even after storage in rapeseed oil for 40 days. An increased OTR was observed in both the PP and HDPE films stored for 40 days in rapeseed oil. The OTR of the HDPE films was altered to a much greater extent than the OTR of the PP film. The OTR of the HDPE film was also significantly increased for films stored for 20 days in rapeseed oil. The increase in OTR of an HDPE film stored for 40 days in rapeseed oil was between 36 and 44%, depending on the relative humidity, compared to the OTR through the virginal polymer. The amount of oil sorbed by the polymer films used decreased in the following order: HDPE » APET > PP.

L15 ANSWER 10 OF 10 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 1992:429756 CAPLUS
DOCUMENT NUMBER: 117:29756
TITLE: Oxygen adsorbents
INVENTOR(S): Shinohara, Kazuhiko
PATENT ASSIGNEE(S): Powder Tech K. K., Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 4 pp.

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                         CODEN: JKXXAF
DOCUMENT TYPE:           Patent
LANGUAGE:                Japanese
FAMILY ACC. NUM. COUNT:  1
PATENT INFORMATION:

    PATENT NO.          KIND   DATE       APPLICATION NO.        DATE
    ---------------     ----   --------   --------------------   --------
    JP 04074528          A     19920309   JP 1990-187070         19900717
PRIORITY APPLN. INFO.:                    JP 1990-187070         19900717
AB  The composite adsorbents comprise an O-adsorbing component from a mixture of
    metal powder, metal halide, and oil and an O-adsorbing component containing
    water, which are packed in a gas-permeable material.  The adsorbents are
    useful for packing food under O-free conditions.


=> d his

    (FILE 'HOME' ENTERED AT 16:51:32 ON 16 JAN 2008)

    FILE 'CAPLUS' ENTERED AT 16:51:59 ON 16 JAN 2008
L1            9 S ORGANIC (L) OZONIDES (L) (MAKE OR PRODUCE)
L2          203 S OZONIZED (L) (FAT? OR OIL?) (L) WATER
L3            0 S L2 AND (PRODUCE OR MAKE) (L) PEROXICE
L4            3 S L2 AND (PRODUCE OR MAKE) (L) PEROXIDE
L5            0 S (OIL AJD WATER) (L) (OZONE OR OXYGEN) AND PEROXIDE
L6          268 S (OIL (4W) WATER) (S) (OXYGEN OR OZONE)
L7           16 S L6 AND PEROXIDE
L8           16 S L7 NOT L4 NOT L1
L9            7 S L8 AND (PROCESS OR MAKE)
L10         852 S (FAT# OR OIL#) (S) WATER (S) (OZONE OR OXYGEN)
L11           2 S L10 AND (PEROXIDE (5W) PRODUCT)
L12           0 S (SUNFLOWER ADJ OIL)(S) WATER (S) (OZONE OR OXYTEN)
L13          11 S (VEGETABLE (A) OIL) (S) WATER (S) (OZONE OR OXYGEN)
L14           0 S L13 NOT L10
L15          10 S L13 NOT L8

=> s (sunflower (A) oil) (S) water (S) (ozone or oxygen)
        27978 SUNFLOWER
         2385 SUNFLOWERS
        28396 SUNFLOWER
                (SUNFLOWER OR SUNFLOWERS)
       807480 OIL
       386515 OILS
       912810 OIL
                (OIL OR OILS)
      2650614 WATER
       271094 WATERS
      2708560 WATER
                (WATER OR WATERS)
        87996 OZONE
           47 OZONES
        88004 OZONE
                (OZONE OR OZONES)
       806169 OXYGEN
         7275 OXYGENS
       811237 OXYGEN
                (OXYGEN OR OXYGENS)
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L16          1 (SUNFLOWER (A) OIL) (S) WATER (S) (OZONE OR OXYGEN)

=> d l16 ibib abs

L16  ANSWER 1 OF 1  CAPLUS  COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER:         1998:749691  CAPLUS
DOCUMENT NUMBER:          129:318216
TITLE:                    Methods for control of oxygen and water levels in
                          supercritical extraction solvents
INVENTOR(S):              Cocero Alonso, M. Jose; Calvo Garrido, Lourdes;
                          Fernandez Polanco, Fernando; Diez Andres, Jose Manuel
PATENT ASSIGNEE(S):       Universidad de Valladolid, Spain
SOURCE:                   Span., 10 pp.
                          CODEN: SPXXAD
DOCUMENT TYPE:            Patent
LANGUAGE:                 Spanish
FAMILY ACC. NUM. COUNT:   1
PATENT INFORMATION:

     PATENT NO.          KIND   DATE       APPLICATION NO.         DATE
     ---------------     ----   --------   --------------------    --------
     ES 2109139           A1    19980101   ES 1994-972             19940503
     ES 2109139           B1    19980816
PRIORITY APPLN. INFO.:                     ES 1994-972             19940503
AB   The method consists on use of a drain cock to eliminate air from
     extractant prior to the extraction and an inlet for feeding antioxidant into
     com. supercrit. extraction and/or supercrit. fluid chromatog. equipment.  The
     antioxidant for oxygen control can be introduced under continuous or batch
     mode, and air is removed prior to addition of antioxidant.  Thus, in
extraction of
     sunflower seed oil with 154 g supercrit. CO2 (99.5%) at 30 MPa and
     40°, the amount of O in the CO2 is 0.38 g which is sufficient to turn
     the oil into rancid oil; When the CO2 purity is 99.999%, i.e., O content
     is 2 ppm and ascorbic acid is added to the oil as antioxidant, the oil
     shows the same stability as that of oil that has not been in contact with
     CO2 or O.  Water presents similar problems in the same type of supercrit.
     extraction of sunflower seed oil.


=> d his

     (FILE 'HOME' ENTERED AT 16:51:32 ON 16 JAN 2008)

     FILE 'CAPLUS' ENTERED AT 16:51:59 ON 16 JAN 2008
L1           9 S ORGANIC (L) OZONIDES (L) (MAKE OR PRODUCE)
L2         203 S OZONIZED (L) (FAT? OR OIL?) (L) WATER
L3           0 S L2 AND (PRODUCE OR MAKE) (L) PEROXICE
L4           3 S L2 AND (PRODUCE OR MAKE) (L) PEROXIDE
L5           0 S (OIL AJD WATER) (L) (OZONE OR OXYGEN) AND PEROXIDE
L6         268 S (OIL (4W) WATER) (S) (OXYGEN OR OZONE)
L7          16 S L6 AND PEROXIDE
L8          16 S L7 NOT L4 NOT L1
L9           7 S L8 AND (PROCESS OR MAKE)
L10        852 S (FAT# OR OIL#) (S) WATER (S) (OZONE OR OXYGEN)
L11          2 S L10 AND (PEROXIDE (5W) PRODUCT)
L12          0 S (SUNFLOWER ADJ OIL)(S) WATER (S) (OZONE OR OXYTEN)
L13         11 S (VEGETABLE (A) OIL) (S) WATER (S) (OZONE OR OXYGEN)
L14          0 S L13 NOT L10
```

```
L15          10 S L13 NOT L8
L16           1 S (SUNFLOWER (A) OIL) (S) WATER (S) (OZONE OR OXYGEN)

=> s ozonized (S) (fat# or oil) and peroxide
          5658 OZONIZED
        309772 FAT#
        807480 OIL
        386515 OILS
        912810 OIL
                 (OIL OR OILS)
           341 OZONIZED (S) (FAT# OR OIL)
        225166 PEROXIDE
         48785 PEROXIDES
        244405 PEROXIDE
                 (PEROXIDE OR PEROXIDES)
L17          47 OZONIZED (S) (FAT# OR OIL) AND PEROXIDE

=> s l17 not l13
L18          47 L17 NOT L13

=> s l17 not l7
L19          47 L17 NOT L7

=> s l19 not l15
L20          47 L19 NOT L15

=> s l20 and water
       2650614 WATER
        271094 WATERS
       2708560 WATER
                 (WATER OR WATERS)
L21          13 L20 AND WATER

=> d l21 1-13 ibib abs
```

L21 ANSWER 1 OF 13 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER:	2006:1314093 CAPLUS
DOCUMENT NUMBER:	146:44462
TITLE:	Chemical analysis of ozonized theobroma fat
AUTHOR(S):	Gomez, Maritza F. Diaz; Tellez, Goitybell Martinez; Cruz, Maikel Arteaga; Mancheno, Rafael Garces
CORPORATE SOURCE:	Department of Ozonized Substances, Ozone Research Center, National Center for Scientific Research, Havana, Cuba
SOURCE:	Journal of the American Oil Chemists' Society (2006), 83(11), 943-946 CODEN: JAOCA7; ISSN: 0003-021X
PUBLISHER:	AOCS Press
DOCUMENT TYPE:	Journal
LANGUAGE:	English

AB Ozonized theobroma fat is used as raw material in the manufacture of pessaries and cosmetic creams. Ozonization of theobroma fat with water was carried out at different applied ozone dosages, and the resultant PV, acid value, iodine value, total hydroperoxide content, and FA content were determined PV and total hydroperoxide content showed a notable increase with applied ozone dosage up to 35.7 mg/g. Acid value varied slightly from 4.1 to 9.9 mg KOH/g, and the iodine value fell

to zero. PV and total hydroperoxide content increased slightly with a higher applied ozone dosage. The comparison of total hydroperoxide measurement using ferrous oxidation in xylenol orange assay and traditional iodometric assay for PV determination showed a significant linear correlation. Small amts. of oleic acid were found in ozonized theobroma fat samples with iodine value equaling zero, which demonstrated that iodine value determination is an inexact assay. During ozonization of theobroma fat, an increase in acid value of 18.9-fold with respect to the initial value was observed owing to decomposition of peroxide.

REFERENCE COUNT: 24 THERE ARE 24 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L21 ANSWER 2 OF 13 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 2006:686197 CAPLUS
DOCUMENT NUMBER: 145:229548
TITLE: In vitro antimicrobial activity of ozonized theobroma oil against Candida albicans
AUTHOR(S): Torres, Iran Fernandez; Pinol, Vicente Curtiellas; Urrutia, Elaine Sanchez; Regueiferos, Magali Gomez
CORPORATE SOURCE: Ozone Research Center, National Center for Scientific Research, Playa. C. Havana, Cuba
SOURCE: Ozone: Science & Engineering (2006), 28(3), 187-190
CODEN: OZSEDS; ISSN: 0191-9512
PUBLISHER: Taylor & Francis, Inc.
DOCUMENT TYPE: Journal
LANGUAGE: English

AB A study of antimicrobial activity of ozonized theobroma oil against Candida albicans was carried out. Min. inhibitory concentration and min. fungicide concentration of ozonized theobroma oil for several peroxides indexes were determined Mortality kinetics for peroxide index of 1200 mmol-eq/Kg was carried out. Effect of contact time, peroxide index and ozonized theobroma oil concentration on Candida albicans mortality was studied. Under the conditions studied, min. inhibitory concentration and min. fungicide concns. 5-3.75 mg/mL and 11.58-5.78 mg/mL were obtained for peroxides indexes of 1002 and 1200 mmol-eq/Kg of sample, resp. Microorganism inactivation follows 1st order kinetics. An optimal concentration of 93.75 mg/mL was obtained. Further increases of ozonized theobroma oil concentration did not exert any effect on mortality. Under the variables ranges studied all parameters have a significant effect on the mortality of Candida albicans, contact time being the most important factor.

REFERENCE COUNT: 12 THERE ARE 12 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L21 ANSWER 3 OF 13 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 2006:354464 CAPLUS
DOCUMENT NUMBER: 144:349310
TITLE: Comparative study of ozonized olive oil and ozonized sunflower oil
AUTHOR(S): Diaz, Maritza F.; Hernandez, Rebeca; Martinez, Goitybell; Vidal, Genny; Gomez, Magali; Fernandez, Harold; Garces, Rafael
CORPORATE SOURCE: Department of Ozonized Substances, Ozone Research Center, National Center for Scientific Research, Havana, Cuba

SOURCE: Journal of the Brazilian Chemical Society (2006), 17(2), 403-407
CODEN: JOCSET; ISSN: 0103-5053
PUBLISHER: Sociedade Brasileira de Quimica
DOCUMENT TYPE: Journal
LANGUAGE: English

AB In this study the ozonized olive and sunflower oils are chemical and microbiol. compared. These oils were introduced into a reactor with bubbling ozone gas in a water bath at room temperature until they were solidified. The peroxide, acidity and iodine values along with antimicrobial activity were determined Ozonization effects on the fatty acid composition of these oils were analyzed using gas-liquid chromatog. technique. An increase in peroxidn. and acidity values was observed in both oils but they were higher in ozonized sunflower oil. Iodine value was zero in ozonized olive oil whereas in ozonized sunflower was 8.8 g Iodine per 100 g. The antimicrobial activity was similar for both ozonized oils except for Min. Bactericidal Concns. of Pseudomonas aeruginosa. Composition of fatty acids in both ozonized oils showed gradual decrease in unsatd. fatty acids (C18:1, C18:2) with gradual increase in ozone doses.

REFERENCE COUNT: 28 THERE ARE 28 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L21 ANSWER 4 OF 13 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 2005:1345817 CAPLUS
DOCUMENT NUMBER: 144:74837
TITLE: Method for suppressing atypical pneumonia coronavirus
INVENTOR(S): Pedder, V. V.; Rot, G. Z.; Obukhova, L. A.; Dushechkina, M. A.; Gaponenko, G. E.; Shataev, A. I.; Temerev, V. L.; Tereshchenko, A. Yu.; Kosenok, V. K.; Shkuro, Yu. V.; Pedder, A. V.
PATENT ASSIGNEE(S): Russia
SOURCE: Russ., 10 pp.
CODEN: RUXXE7
DOCUMENT TYPE: Patent
LANGUAGE: Russian
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
RU 2266757	C2	20051227	RU 2003-121747	20030714
PRIORITY APPLN. INFO.:			RU 2003-121747	20030714

AB The method involves administering ultrasonic ozonide-containing drug aerosol inhalation as ozonized oil and water mixture Ultrasonic inhalation is addnl. applied to bronchopulmonary system using highly refined aerosol containing ozonized oil having ozonide-based peroxide number P=800-900 and aerosol particle diameter equal to 0.3-0.5 mcm, giving at least two sessions per day at pathol. process active phase stage. Bronchopulmonary system tissue edema being relieved, ultrasonic inhalation procedure is first applied using highly refined aerosol containing ozonized oil having 20% ozonized oil emulsion of oil-in-water type having peroxide number P=200 and aerosol particle diameter equal to 0.3-0.5 mcm, giving at least two sessions per day at infection regeneration stage until clin. proved patient recovery data being obtained. Next, ultrasonic inhalation procedure is applied using medium

refined aerosol containing ozonized oil having
peroxide number P=800-900 and aerosol particle diameter equal to
5.0-10.0 mcm, each inhalation procedure being at least 10 min long.EFFECT:
enhanced effectiveness of treatment.

L21 ANSWER 5 OF 13 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 2005:452597 CAPLUS
DOCUMENT NUMBER: 143:171668
TITLE: Study of three systems of ozonized coconut oil
AUTHOR(S): Diaz, Maritza F.; Nunez, Nayibi; Quincose, Daraisy; Diaz, Wilfredo; Hernandez, Frank
CORPORATE SOURCE: Ozone Research Center, National Center for Scientific Research, Havana, Cuba
SOURCE: Ozone: Science & Engineering (2005), 27(2), 153-157
CODEN: OZSEDS; ISSN: 0191-9512
PUBLISHER: Taylor & Francis, Inc.
DOCUMENT TYPE: Journal
LANGUAGE: English
AB Products from the ozonization of 3 systems (ozonized coconut oil, ozonized coconut oil with water and ozonized coconut oil with ethanol) are characterized and compared statistically. Peroxide, acidity and viscosity indexes were determined The reaction products were identified by 1H NMR and their antimicrobial activity was evaluated. The ozonized coconut oil with ethanol showed the highest peroxide and acidity indexes. This result suggests that in presence of ethanol a greater peroxide decomposition occurs leading to greater acid formation. The variation coeffs. obtained in the anal. methods were <10%. Reaction products were identified as ozonides and aldehydes compds. The highest action spectrum of antimicrobial activity by Staphylococcus aureus was obtained with the ozonized coconut oil with water and ethanol systems.
REFERENCE COUNT: 23 THERE ARE 23 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L21 ANSWER 6 OF 13 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 2003:428847 CAPLUS
DOCUMENT NUMBER: 139:90408
TITLE: Method for suppressing HIV infection
INVENTOR(S): Pedder, V. V.; Sergienko, G. G.; Lyutvina, E. G.; Temerev, V. L.; Pedder, A. V.; Legostaeva, N. A.
PATENT ASSIGNEE(S): Russia
SOURCE: Russ., No pp. given
CODEN: RUXXE7
DOCUMENT TYPE: Patent
LANGUAGE: Russian
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
RU 2198664	C2	20030220	RU 1999-123816	19991112

PRIORITY APPLN. INFO.: RU 1999-123816 19991112
AB One should perform ultrasound sanitation of emptied rectum. Then comes gradual ultrasound impregnation of ozonized physiol. solution or ozonized fatty emulsion. It is necessary to conduct ultrasound contact rectal treatment. Then one should carry out endovaginal, and in men - urethral

impregnation of ultrasound aerosol of medicinal preparation into mucous tissues followed by aeration with either ozone-oxygen or ozone-air gaseous mixts. As ozonized physiol. solution one should use 0.9% sodium chloride solution or 5% glucose solution Ozone concentration in solution corresponds to 1-10 mg/L. Ozone concentration in ozone-oxygen and ozone-air gaseous mixture is 1-20 mg/L. Peroxide number P of ozonized fatty emulsion of oil -in-water type is about 500, not lower. The present method enables to maintain body ozone dosage for the period exceeding, at least, one HIV replication cycle.

L21 ANSWER 7 OF 13 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 2001:348032 CAPLUS
DOCUMENT NUMBER: 135:111587
TITLE: Volatile components of ozonized sunflower oil "OLEOZON"
AUTHOR(S): Ledea, Oscar; Correa, Teresa; Escobar, Mayhery; Rosado, Aristides; Molerio, Jesus; Hernandez, Carlos; Jardines, Daniel
CORPORATE SOURCE: Ozone Research Center, National Center for Scientific Research, Havana City, Cuba
SOURCE: Ozone: Science & Engineering (2001), 23(2), 121-126
CODEN: OZSEDS; ISSN: 0191-9512
PUBLISHER: Lewis Publishers
DOCUMENT TYPE: Journal
LANGUAGE: English
AB During the ozonization of the triglycerides and unsatd. fatty acids from sunflower oil, aldehydes and carboxylic acids with 3, 6 and 9 C atoms are obtained, together with hydroperoxides, ozonides and other peroxide species. Some of them present relatively low b.ps., constituting the volatile fraction of OLEOZON. The volatile fraction was characterized by gas chromatog.-mass spectrometry (GC/MS) and GC combined with 3 different extraction techniques: gaseous purge with cold trap collection, gaseous purge with Tenax adsorption followed by a solvent elution and liquid-liquid extraction The volatile fraction of OLEOZON is composed by saturated and unsatd. aldehydes and carboxylic acids. Hexanal, nonanal, 3-nonenal and malonaldehyde were the main components of the volatile fraction.
REFERENCE COUNT: 18 THERE ARE 18 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L21 ANSWER 8 OF 13 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 1969:77339 CAPLUS
DOCUMENT NUMBER: 70:77339
ORIGINAL REFERENCE NO.: 70:14409a
TITLE: Acetal peroxides
INVENTOR(S): Gabelein, Klaus
PATENT ASSIGNEE(S): Hentschke und Sawatzki Chemische Fabrik
SOURCE: Brit., 4 pp.
CODEN: BRXXAA
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE

GB 1138075		19681227	GB 1966-2429	19660119
PRIORITY APPLN. INFO.:			DE	19650122

AB Unsatd. compds. are treated with ozone and alc., and poly(oxyethylene) (I) derivs., at <70° to give the title compds. A solution is prepared at 60° from 10 g. sorbic acid and 100 ml. I (mol. weight .apprx.400) mono ether, cooled to 20°, and ozonized to give an oil which is miscible with water and alc. Similarly prepared are (olefin reactant and alc. given): castor oil, dodecanol; castor oil; I (mol. weight 600); olive oil, stearyl alc.; cedarwood oil, isotridecan-2-ol; oleyl alc., and PhCH2OH.

L21 ANSWER 9 OF 13 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 1963:461678 CAPLUS
DOCUMENT NUMBER: 59:61678
ORIGINAL REFERENCE NO.: 59:11281a-h,11282a-b
TITLE: Constituents of Umbelliferae. VII. Structure of ligustilide. 2
AUTHOR(S): Mitsuhashi, H.; Nagai, U.
CORPORATE SOURCE: Hokkaido Univ., Sapporo
SOURCE: Tetrahedron (1963), 19(8), 1277-83
CODEN: TETRAB; ISSN: 0040-4020
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
GI For diagram(s), see printed CA Issue.
AB cf. CA 55, 23690i; 57, 16744e. Isolation from Cnidium officinale gave better yields of the previously isolated ligustilide (I). I (6.8 g.) in 45 ml. C6H14 hydrogenated over 0.7 g. 5% Pd-BaCO3 at 15°/760 mm. with adsorption of 410 ml. H the filtered solution evaporated, the residual oil (6.0 g.) chromatographed on 200 g. silicic acid (Mallinkrodt 100 mesh) and eluted with CHCl3, the fractions (γ 276 mμ, CHCl3) collected, and the solvent evaporated in vacuo gave 3.1 g. product, rechromatographed to yield dihydroligustilide (II), γ 272 mμ (log ε 4.25), ν 1755, 1685, 1647 cm.-1 (neat), together with small amts. of tetrahydroligustilide (III) and 3-butylphthalide. II (2.3 g.) in 50 ml. CHCl3 ozonized 2 hrs. in a stream of 3% ozonized O, the residue on evaporation stirred 1 hr. (ice bath) with 1.5 g. NaOH and 3 ml. 30% H2O2 in 20 ml. H2O, the mixture treated with 3 ml. 30% H2O2 and warmed 1 hr. at 60°, kept 16 hrs. at 20°, acidified, steam distilled, the distillate (100 ml.) titrated with N NaOH, and the neutral solution evaporated gave 0.8 g. residue, partially subjected to paper chromatographic analysis in BuOH saturated with 1.5N NH4OH. The remainder of the above residue was acidified and extracted continuously with Et2O, and the extract concentrated and subjected to gas chromatographic analysis. Both analyses showed the presence of PrCO2H and a trace of HCO2H. The steam distillation residue extracted with Et2O and the product (1.9 g.) recrystd. from Et2O gave 0.7 g. adipic acid, m. 150-1°. Traces of glutaric and succinic acids were also detected by paper chromatography. II (30.7 g.) refluxed 1 hr. in 500 ml. alc. 5% KOH, the mixture diluted with 500 ml. H2O, the solution concentrated with passage of 500 ml. distillate, and the aqueous alkaline solution washed with Et2O, saturated with CO2, washed with Et2O, acidified, and extracted with Et2O gave 24.9 g. acid fraction, crystallized from 1: 10 EtOAcC6H14 to yield 9.2 g. IV, recrystd. from 1:5 EtOAc-C6H14 to yield 19.3%, m. 108-10° (5.4 g.) and 105-8° (1.1 g.). IV treated with N2H4. H2O gave the phthalazone (V), γ 282 mμ (ε 3770), assigned the given structure by comparison with 3-pyridazone, γ 280 mμ

(ε 3550). I (490 mg.) in C6H14 hydrogenated at 20°/760 mm.
over 100 mg. 5% Pd-C until 104 ml. H was taken up, the filtered solution evaporated, and the residual oil chromatographed on 10 g. silicic acid and eluted with CHCl3 gave 290 mg. III, γ 217 mμ (log ε 4.08), ν 1740, 1675 cm.-1 (CHCl3), yielding an oxo acid on hydrolysis and requiring the presence of an α,β-unsatd. butenolide moiety. The nuclear magnetic resonance (n.m.r.) spectrum of I indicated the presence of a butylidene side chain and 3 olefinic protons. The assigned structure fulfills these requirements and explains the strong ultraviolet absorption at 320 mμ. Maleic anhydride (15.0 g.) in 100 ml. warm xylene and 3.0 g. I saturated with N and heated 28 hrs. at 130-50° (oil bath) in a thick-walled sealed glass tube, xylene and excess anhydride evaporated in vacuo, and the residue recrystd. twice from C6H6 yielded 0.7 g. crystalline adduct (VI), m. 164-6°, γ 221 mμ (log ε 3.93), ν 1860, 1840-1825, 1770, 1705, 1645 cm.-1 VI (0.8 g.) refluxed 2 hrs. in 30 ml. 5% alc. KOH, the alc. replaced by H2O, the alkaline aqueous solution washed with Et2O, acidified, extracted continuously with Et2O 16 hrs., the crystals separating out treated with hot EtOAc, and the soluble fraction recrystd. from EtOAc gave 0.3 g. oxo acid (VII), m. 180-2° (decomposition), γ 214 mμ (log ε 3.82), ν 3620-3550, 1780, 1710-1690, 1618 cm.-1 The insol. fraction recrystd. from Me2CO gave 0.1 g. acid (VIII), m. 162-4° (decomposition), γ 211 mμ (log ε 3.79), ν 3560, 1710-1690, 1615 cm.-1 The Et2O residue (0.6 g.) recrystd. from C6H6 gave 0.1 g. acid (IX), m. 230° (decomposition), γ 214 mμ (log ε 3.91), ν 3500-3350, 1728, 1705, 1683, 1618 cm.-1 The acids VII, VIII, and IX taken up in min. H2O, treated with 3 equivs. of K2CO3, and the neutral solns. evaporated in vacuo and dried in an evacuated desiccator over CaCl2 gave soap-like adduct-hydrolyzates as tri-K salts. VII (130 mg.) in 15 ml. EtOAc streamed through 1.5 hrs. with 3.5% ozonized O, the solution extracted with 5% aqueous NaHCO3 containing 10% H2O2, the alkaline solution kept 1 hr. at 20° and heated 15 min. on a boiling water bath, the solution acidified, kept 16 hrs., treated dropwise with Na2S2O4 (to neg. peroxide test with KI) and centrifuged, the S-free supernatant liquid extracted with Et2O, the product (85 mg.) subjected to partition chromatography on a column prepared with 20 g. silicic acid, 12 ml. H2O, and 80 ml. CHCl3, the sample charged in CHCl3-BuOH and eluted with 100 ml. mixts. of CHCl3-BuOH (100:0, 19:1, 9:1, 17:3, 4:1, 3:1, 7:3, 3:2, and 1:1), 10 ml. fractions collected, fractions 21-3 combined and evaporated, and the residue (19 mg.) recrystd. 3 times from EtOAc gave 3 mg. acid (X), C14H20O7, m. 185-7°, 1720-1700 cm.-1 The acids VII, VIII, and IX are considered to be diastereoisomeric with respect to the configuration of the CO2H group, which can be epimerized under the conditions of alkaline hydrolysis and are tautomeric concerning the conversion of the oxo acid to the lactonol. Possible structures of the oxo acids were charted. VI was considered the correct structure for the adduct and was confirmed by ozonolysis of VII to X. The problem of double bond migration during the reactions was discussed.

L21 ANSWER 10 OF 13 CAPLUS

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TITLE: Double-bond isomerization in higher-molecular olefins. IV. The bond-isomerizing effect of various compounds on n-undecenes

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AB cf. CA 37, 46841,2·; 50, 7047f. The double-bond isomerization in 1-(I) and 5-undecene (II) by acidic, basic, and neutral compds. was investigated. A particularly effective isomerization catalyst is Fe(CO)5 (III) decomposing thermally or under ultraviolet irradiation. The quant. analysis of the isomeric n-undecenes was performed by oxidative ozonolysis and gas chromatography of the resulting carboxylic acids as their Me esters. CH2:CHCH2Br (266 g.) in 800 cc. dry Et2O added dropwise with stirring and cooling to C8H17MgCl from 60 g. Mg and 327 g. C8H17Cl in 1200 cc. dry Et2O, refluxed 2 hrs. with stirring, poured onto 1-2 kg. crushed ice, and worked up yielded I, b13 74.5-6.0°, b755 191.5-2.5° n20D 1.4262, containing 0.2% II, 2.7% 2-undecene (IV), 0.8% 3-undecene (V), and 0.2% 4-undecene (VI). A series of 4 similar runs was performed to yield I of 93.1, 96.3, 97.1, and 95.1% purity, resp. AmOH with SOCl2 in C5H5N yielded 30% AmCl, b736 107-7.7°, n20D 1.4128-1.4129. AmCHO (101 g.) in 200 cc. dry Et2O added dropwise to AmMgCl from 118 g. AmCl and 29 g. Mg in 600 cc. dry Et2O, poured onto 250 g. ice, treated with 200 cc. dilute HCl, and worked up yielded 117 g. Am2CHOH (VII), b12 112.5-12.8°, n25D 1.4345, m. 24.8-5.8°; 2,5-dinitrobenzoate m. 48°. VII (205 g.) added dropwise during 0.5 hr. at 100° to 333 g. C17H35COCl, stirred 3 hrs. at 120°, cooled to 80°, treated with 5 cc. MeOH, heated slowly to 290° while being treated with N and then to 320°, and the distillate fractionated yielded 125 g. II, b724 190.5-1.5°, n20D 1.4293, containing 1.5% VI. The isomeric undecenes (2 g.) in 25 cc. hexane ozonized about 15 min. at -15° with about 25 l. O containing 4 volume-% ozone/hr., added at 90° to 15 g. Ag2O in 70 cc. 10% aqueous NaOH during 10 min. with stirring, stirred 3 hrs. on the water bath, cooled to 50°, treated dropwise with stirring during 5 min. with 43 cc. 65% HNO3, cooled to room temperature, and extracted with pentane, and the extract concentrated gave the carboxylic acids; the crude product dissolved in 100 cc. 4% KOH in 1:1 MeOH-H2O, washed with C6H6 and petr. ether, the washings reextd. with 4% KOH in 1:1 MeOH-H2O, the combined alkaline exts. acidified with 65% HNO3 and extracted with pentane, and the pentane extract treated with CH2N2 from about 3 g. H2NCON(NO)Me in pentane gave the corresponding Me esters which were gas-chromatographed. The compds. measured were C9H19CO2Me, C8H17CO2Me, C7H15CO2Me, C6H13CO2Me, and AmCO2Me for I, IV, V, VI, and II, resp. I (3.0 g.) in 35 cc. pentane ozonized at -15° during 1 hr. under the usual conditions and worked up showed that the lower homologs in the resulting acid mixture were enriched only by about 3.5%. Ozonization at -60° (performed in the same manner) resulted in incomplete reaction. I (4.5 g.) and 0.29 g. Li, or 1.0 g. Na, or 1.7 g. K refluxed with stirring gave a suspension of the alkali metal; the isomerization runs with the alkali metals were carried out with 40 mole-% I and 60 g.-atom % alkali metal in the absence of an added halo compound and with 38 mole-% I and 57 g.-atom % metal in the presence of 5 mole-% halo compound; after completion of the run, the mixture was filtered, and the filtrate distilled to give the isomer mixture which was analyzed in the usual manner. By this procedure were performed the following runs (catalyst, added halo compound, reaction time in hrs., % purity of I used, and % content of I, IV, V, VI, and II in the resulting isomer mixture given): Li, none, 5, 96.3,

91.8, 5.1, 1.1, 1.1, 0.9; Li, C8H17Cl, 5, 96.3, 92.1, 5.6, 1.1, 0.6, 0.5; Li, PhCl, 2, 96.3, 92.6, 5.2, 1.0, 0.6, 0.5; Na, none, 5, 93.4, 90.2, 8.2, 1.0, 0.4, 0.3; Na, C8H17Cl (1.8 mole %), 5, 95.3, 90.4, 7.3, 1.1, 0.7, 0.5; Na, C8H17Cl (5 mole %), 5, 93.1, 67.0, 30.4, 1.6, 0.6, 0.4; Na, PhCl, 5, 96.3, 2.7, 32.2, 29.1, 24.0, 12.0; Na, PhCl, 0.5, 96.3, 2.1, 31.5, 28.5, 24.9, 13.0; Na, PhBr, 5, 96.3, 4.5, 32.4, 27.2, 22.9, 13.1; Na, o-MeC6H4Cl, 5, 96.3, 1.3, 28.0, 26.6, 26.9, 17.2; Na, m-MeC6H4Cl, 5, 96.3, 1.5, 28.3, 26.4, 27.0, 16.8; Na, CH2:CHCH2Cl, 2, 96.3, 93.9, 4.8, 0.7, 0.3, 0.3; Na, p-MeC6H4Cl, 5, 96.3, 3.6, 31.8, 27.3, 23.6, 13.6; K, none, 0.5, 96.3, 91.8, 6.2, 1.2, 0.5, 0.3; K, C8H17Cl, 0.5, 96.3, 89.3, 6.9, 1.4, 1.7, 0.7; K, PhCl, 0.5, 96.3, 13.1, 65.8, 16.5, 3.6, 1.1. A similar run with 5 mole-% 0.7M PhLi refluxed 5 hrs. under Ar (after removal of the Et2O) gave from 97.1%-pure I a mixture containing 92.2 I, 1.5 IV, 0.4 V, 0.4 VI, and 0.4% II. I (3.0 g.) (97.1%-pure) refluxed 5 hrs. with 5 mole % PhLi in 20 cc. dry Et2O and decomposed with H2O, and the organic phase worked up yielded a mixture containing 97.2 I, 1.8 IV, 0.3 V, 0.3 VI, and 0.4% II. A run with 97.1%-pure I and 50% BuLi at 25° under Ar during 24 hrs. gave a mixture of 96.7 I, 2.4 IV, 0.5 V, 0.4% VI, and a trace of II. Na (about 3 g.) as 10 cc. 33% Na dust dispersion in xylene, 30 cc. MePh, and 5.5 g. dry PhCl stirred and treated with pure Ar during 1 hr. at 35-40°, the mixture treated with 7.7 g. 97.1%-pure I, stirred 1 hr. at 50° under Ar, poured onto powdered dry ice in portions, diluted with H2O, and acidified gave 5.7 g. BzOH; the organic phase dried and distilled gave a mixture containing 3.8 I, 85.6 IV, 8.5 V, 1.4 VI, and 0.7% II. A run with II performed during 0.5 hr. with Na and PhCl in the usual manner gave a mixture containing 0.2 I, 4.2 IV, 11.4 V, 36.7 VI, and 47.5% II. All alkali alcoholates used for the isomerization runs were obtained by dissolving the alkali metal in a large excess of the appropriate alc., mixing an aliquot containing 5 mole-% and adding to the I and then removing the excess alc. with the exception of C8H17OH. (BuO)2Ca was obtained similarly by refluxing Ca in BuOH for 12 hrs. B(OAc)8 from B(OH)3 and Ac2O transesterified with the equivalent amount appropriate alc. yielded the corresponding B(OR)3 (R, % yield, b.p./mm., and n20D given): Pr (VIII), 22, 74-84°/13, 1.3954; Bu (IX), 40, 109-14°/13, 1.4094; iso-Bu (X), 38, 105-10°/19, 1.4033; EtMeCH (XI), 32, 88-95°/19, 1.3972; Am (XII), 48, 146-51°/16, 1.4190. TiCl4 treated with NaOEt in absolute EtOH yielded 10% Ti(OEt)4 (XIII), b13 154-4.5°. I (3.0 g.) and 5 mole-% appropriate alcoholate or ester refluxed 5 hrs. at 190° and distilled gave the isomer mixture A series of runs was performed in this manner (alcoholate or ester used as catalyst, % purity of I used, and % content of I, IV, V, VI, and II in the resulting isomer mixture given): BuOLi, 96.3, 94.8, 4.0, 0.7, 0.3, 0.2; EtMeCHOLi, 96.3, 96.8, 2.4, 0.4, 0.3, 0.2; tert-BuOLi, 96.3, 96.7, 2.6, 0.3, 0.3, 0.1; C8H17OLi, 96.3, 93.8, 2.8, 2.8, 0.5, 0.1; BuONa, 96.3, 95.8, 3.1, 0.6, 0.3, 0.2; EtMeCHONa, 96.3, 96.4, 2.2, 0.6, 0.3, 0.5; tert-BuONa, 96.3, 96.9, 2.0, 0.4, 0.3, 0.4; C8H17ONa, 93.1, 90.2, 8.2, 1.0, 0.4, 0.3; BuOK, 96.3, 78.0, 17.9, 2.7, 0.9, 0.4; EtMeCHOK, 96.3, 42.5, 54.7, 1.8, 0.6, 0.3; EtMeCHOK, 96.3, 47.1, 49.7, 2.2, 0.7, 0.4; EtMeCHOK, 96.3, 62.2, 36.1, 1.2, 0.4, 0.2; tert-BuOK, 96.3, 72.7, 24.1, 1.7, 1.0, 0.6; tert-BuOK (in tert-BuOH), 96.3, 74.2, 21.9, 2.4, 0.9, 0.6; C8H17OK, 96.3, 93.5, 4.8, 1.2, 0.3, 0.2; C8H17OK, 96.3, 91.5, 6.4, 1.6, 0.3, 0.2; C8H17OK, 96.3, 89.9, 6.7, 1.9, 0.9, 0.4; 20% KOH-(CH2OH)2 (25 mole %) (at 180°), 96.3, 94.3, 3.8, 0.8, 0.4, 0.6; (BuO)2Ca, 96.3, 93.6, 4.4, 1.0, 0.5, 0.5; (iso-PrO)3Al, 93.1, 91.9, 5.4, 2.2, 0.3, 0.2; (EtMeCHO)3Al, 93.1, 92.8, 5.8, 0.9, 0.3, 0.2; VIII, 93.1, 90.9, 6.8, 1.5, 0.6, 0.3; IX, 93.1, 91.3, 6.7, 1.2, 0.4, 0.3; X, 93.1, 91.3, 6.7, 1.2,

0.4, 0.3; XI, 93.1, 92.4, 5.7, 1.2, 0.5, 0.3; XII, 93.1, 91.7, 6.2, 1.4, 0.5, 0.3; XIII, 93.1, 93.1, 5.3, 1.0, 0.4, 0.2; (BuO)4Ti, 93.1, 93.7, 4.6, 0.8, 0.5, 0.4. A similar run with (iso-PrO)3Al and II (98.5%-pure) gave a mixture of 91.5% II, 1.4% V, and 7.2% VI. I shaken with 10 or 5 mole-% aqueous acid at room temperature, neutralized with Na2CO3, washed with H2O, dried, and distilled gave an isomer mixture; a similar series of runs was performed with added Me2CO to achieve homogeneity; the mixture was kept 1 hr. at room temperature, neutralized with Na2CO3, and diluted with H2O to precipitate the isomer mixture In this manner were prepared the following runs (aqueous catalyst and mole-% used, purity of I used, % content of I, IV, V, VI, and II in the resulting isomer mixture, and % polymer obtained are given): 40% HF, 10, 96.3, 94.7, 3.7, 0.8, 0.4, 0.4, 5; 44% HBF4, 10, 96.3, 93.6, 3.6, 0.8, 0.9, 1.7, --; 70% HClO4, 5, 96.3, 94.8, 3.5, 0.8, 0.5, 0.5, --; 40% HF (and Me2CO), 10, 97.1, 97.4, 1.1, 0.4, 0.5, 0.7, 2; 44% HBF4 (and Me2CO), 10, 97.1, 97.4, 0.9, 0.4, 0.4, 0.8, 2; 70% HClO4 (and Me2CO), 5, 97.1, 96.6, 0.9, 0.4, 0.7, 1.4, 5. I (97.1%-pure) (3.0 g.), 0.17 cc. 70% HClO4, and 10 cc. AcOH heated 5 min. at 100°, diluted with H2O, and worked up in the usual manner yielded a mixture of 93.1 I, 4.8 (IV), 0.9 V, 0.6 VI, 0.7% II, and some undecyl acetate. I (96.3%-pure) (3.0 g.) and 10 mole-% concentrated H2SO4 stirred at -40° during 2 hrs., neutralized with excess aqueous Na2CO3, diluted with MeOH to break the emulsion, and extracted with hexane gave a mixture containing 86.3 I, 9.8 IV, 1.8 V, 1.0 VI, and 1.7% II, and 5% polymer. I (96.3%-pure) and 5 mole-% p-MeC6H4SO3H refluxed 5 hrs. gave 20% isomer mixture and 80% black oil and charred solid as residue; the isomer mixture consisted of 15.7 I, 28.3 IV, 31.9 V, 18.4 VI, and 5.7% II. I (96.3%-pure) (3.0 g.) and 5 mole-% 97-8% PhSO3H refluxed 1 hr. and distilled gave 80% mixture of 1.8 I, 28.5 IV, 28.2 V, 27.7 VI, and 13.9% II, and 20% oily polymer. I (96.3%-pure) (3.0 g.) and 10 mole-% C8H17Cl refluxed 10 hrs. (HCl evolved) and distilled yielded 50% mixture of 5.4 I, 34.4 IV, 28.3 V, 20.0 VI, and 11.8% II, and 50% oily residue. I (95.3%-pure) (3.0 g.) and 0.29 g. C8H17Cl refluxed 10 hrs. and ozonized without the previous removal of the C8H17Cl showed the presence of a mixture of 94.5 I, 3.2 IV, 1.1 V, 0.5 VI, and 0.7% II. I (97.1%-pure) (3.0 g.), 0.29 g. 70% HClO4, and a sufficient amount of Ac2O to remove the H2O kept 0.5 hr. at 100° gave a mixture of 4.0 I, 40.0 IV, 28.1 V, 18.4 VI, and 9.5% II and left 5% oily residue. I (96.3%-pure) (3.0 g.) and 0.201 g. AgClO4 refluxed 1 hr. yielded a mixture of 1.5 I, 26.9 IV, 26.7 V, 27.6 VI, and 17.3% II and left 5% oil. I (96.3%-pure) (3.0 g.) and 0.13 g. AlCl3 refluxed 5 hrs. and decomposed with excess 20% aqueous NaOH, and the organic layer worked up gave only polymer products. A similar run during 0.5 hr. yielded 50% mixture of 92.9 I, 4.6 IV, 1.2 V, 0.7 VI, and 0.7% II and 50% polymer. A similar run during 0.5 hr. in MeNO2 as solvent gave from 3.0 g. 97.1%-pure I a mixture of 78.4 I, 9.4 IV, 5.1 V, 4.1 VI, and 2.9% II and 20% polymer. I (96.3%-pure) (2.0 g.) treated slowly at room temperature with BF3 during 1.5 hrs. gave only polymer. A similar run during 5 min. gave a mixture of 82.3 I, 11.8 IV, 2.6 V, 1.4 VI, and 1.9% II, and 30% polymer. I (96.3%-pure) (3.0 g.) and 0.125 cc. Et2O.BF3 heated 5 hrs. at 120° gave a mixture of 56.7 I, 22.0 IV, 9.9 V, 7.1 VI, and 4.3 II and left 15% oily polymer. I (97.1%-pure) (3.0 g.), 0.125 cc. Et2O.BF3, and 3.0 cc. dry Et2O refluxed 3 hrs. gave a mixture of 97.3 I, 0.8 IV, 0.5 V, 0.6 VI, and 0.8% II and left 2% polymer. I (97.1%-pure) (3.0 g.) and 5 mole-% BF3.2AcOH, b15 64-6°, shaken 1 hr. at 25°, washed with aqueous Na2CO3 and H2O, dried, and distilled yielded a mixture M 96.1 I, 2.2 IV, 0.7 V, 0.5 VI, and 0.5% II and left 5% polymer. ofg (1.0 g.) in 50 cc. dry Et2O treated with 6.4 g. Br, the resulting 2-phase mixture treated with 6.0 g.

93.1%-pure I, kept 48 hrs. and distilled gave a mixture of 92.2 I, 5.6 IV, 1.2 V, 0.6 VI, and 0.5% II. MgBr2.3Et2O (0.04 mole) treated with dioxane, the crystalline dioxane-0.5 MgBr2 dissolved in 50 cc. refluxing PrOH, the solution refluxed 5 hrs. with 6 g. 93.1%-pure I, concentrated to remove the PrOH, filtered, and distilled gave a mixture of 91.8 I, 5.1 IV, 2.5 V, 0.3 VI, and 0.3% VII. MeI (1.42 g.) in Et2O converted with Mg to MeMgI, the Et2O replaced by 14 cc. dry iso-Am2O, a sufficient volume to make 20 mole-% MeI2 + MeMgI added to 6 g. 97.1%-pure I, the mixture heated 5 hrs. under Ar at 120°, decomposed with H2O, and worked up gave a mixture containing 88.4 I, 7.2 IV, 2.0 V, 1.0 VI, and 1.4% II, and left 8% polymer. A series of isomerization runs was performed with 3.0-g. amts. of I (of various purities) and 0.19 g. III at various temps. (in the runs at 190°, the undecene was distilled oil; the runs at lower temps. were ozonized without the previous removal of the III); at 190° the III decomposed with the formation of an Fe mirror, at 100° with the precipitation of a red-brown solid; the runs with simultaneous ultraviolet irradiation were performed in glass equipment under N or Ar. The following runs were performed in this manner (% purity of the I used, reaction temperature, reaction time in hrs., and % content of I, IV, V, VI, and II in the resulting undecene mixture given): 96.3, 20°, 65, 92.0, 5.2, 1.5, 0.8, 0.5; 96.3, 100°, 1, 70.9, 16.4, 6.8, 3.8, 2.1; 93.1, 190°, 0.17, 1.9, 29.9, 26.3, 27.1, 14.9; 93.1, 190, 0.17, 3.1, 25.8, 24.6, 26.4, 20.1; 93.1, 190° (irradiated before heating), 0.17, 2.1, 28.6, 25.0, 26.7, 17.5; 96.3, 190° (irradiated before heating), 0.17, 2.9, 28.6, 25.8, 28.0, 14.7. Similar runs were performed with II (same data given): 98.5, 190°, 0.17, 2.7, 14.3, 13.1, 16.8, 53.2; 98.5, 190°, 0.25, 3.2, 20.7, 19.6, 21.1, 35.5; 98.5, 190°, 0.5, 2.2, 20.2, 18.1, 20.8, 38.7; 98.5 (with 3-fold amount III), 190°, 3 times 0.25, 1.8, 25.9, 22.5, 24.2, 25.6. A series of runs with I was performed under simultaneous irradiation (same data given): 96.3, 50° (360-w. lamp), 5, 50.5, 20.3, 14.2, 9.5, 5.5; 97.1, 25° (360-w. lamp), 5, 70.7, 13.3, 8.2, 5.0, 2.8; 96.3, 25° (460-w. lamp), 5, 8.2, 35.4, 27.1, 19.5, 9.8. III (0.38 g.) shaken several hrs. under Ar at room temperature with 40 cc. 10% aqueous NaOH, the solution treated with 3 g. 97.1%-pure I and acidified dropwise at 0° under Ar with 40 cc. cold 15% H2SO4, the mixture warmed during 1 hr. to 20°, kept 3 hrs. at 20°, and the organic phase worked up gave a mixture of 81.3 I, 12.5 IV, 2.9 V, 2.0 VI, and 1.3% II. III (2.8 cc.) in 12 cc. MeOH shaken under Ar a few min. with 6 cc. 50% aqueous NaOH, the mixture treated with 3.0 g. 97.1%-pure I in 12 cc. MeOH and 24 cc. iso-PrOH, kept 24 hrs. at 15°, and diluted with 60 cc. H2O precipitated a mixture of 58.2 I, 36.4 IV, 2.8 V, 1.5 VI, and 1.1 II. I (96.1%-pure) irradiated 0.5 and 5 hrs. with a 360-w. ultraviolet lamp gave a mixture of 91.7 I, 5.3 IV, 2.7 V, 0.4% II with a trace of II, and a mixture of 92.9 I, 4.9 IV, 1.1 V, 0.6 VI, and 0.5% II, resp. Bz2O2 (93%-pure) dissolved with 25% H2O in CHCl3 and repptd. with MeOH and dried, a 0.246-g. portion dissolved at 50-70° in 3 g. 96.3%-pure I, and the mixture heated 5 hrs. at 190° gave a mixture of 94.9 I, 3.6 IV, 0.6 V, 0.5 VI, and 0.4% II. Ph3CCl (1.4 g.) in 60 cc. dry C6H6 shaken 5 min. with 3.5 g. Zn dust, treated with 3.0 g. 96.3%-pure I, refluxed 5 hrs., shaken with air to convert the radical to the peroxide, concentrated, filtered, and distilled gave a mixture of 93.3 I, 4.7 IV, 1.1 V, 0.5VI, and 0.5% II.

L21 ANSWER 11 OF 13 CAPLUS
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AUTHOR(S): Holmes, R. L.; Moreau, J. P.; O'Connor, R. T.
CORPORATE SOURCE: Southern Regional Research Lab., New Orleans, LA
SOURCE: Journal of the American Oil Chemists' Society (1962), 39, 411-14
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AB Petroselenic acid (20 g.) isolated (CA 54, 25897i) from parsley seed oil is ozonized with a Welsbach T-23 laboratory ozonizer at -10° in 200 ml. MeOH, placed in ice bath, 25 ml. glacial HOAc added, and then slowly 10 g. Zn dust, keeping the temperature at 10-15° till test for peroxides is neg. Zn sludge is removed by filtration, and oximes are prepared by addition of 12.3 g. NH2OH.HCl, followed by 14.5 g. NaOAc, stirring the solution 2 hrs. and allowing to stand overnight. The amount of H2O used to dissolve the above added salts was enough to give a 75-80% MeOH solution The oximes formed are separated by evaporation to dryness at reduced pressure and at below 50°, addition of 200 ml. H2O and 300 ml. ether, making the mixed phases alkaline with 50% NaOH, and extracting each phase 4 times with the other solvent. The water phase after separation of lauraldehyde oxime is made strongly acid with HCl and extracted 8-10 times with like vols. of ether. The combined ether exts. are dried with Na2SO4 and evaporated under reduced pressure to yield crude adipaldehydic acid oxime. Both oximes were reduced in absolute EtOH with Raney Ni according to Adkins and Billica (CA 42, 3328c) except that the catalyst was more thoroughly washed. The redns. were carried out with 10-15 equivs. liquid NH3, 15% catalyst, 1000 lb. pressure, 95-105°, for 4 hrs. and the solvent was evaporated at 50° under reduced pressure. The dodecylamine was purified by precipitation from ether with HCl gas.
The crude ammonium hexanoate was purified by adsorption on Dowex-50 resin, eluted with dilute NH4OH, the ammonium salt absorbed on Dowex 1 resin, the column washed and eluted with dilute HCl, and the eluate passed through IR-4B resin column (to split off HCl), and crystallized (Meyers and Miller, CA 47, 1596h). The infrared spectra of lauraldehyde oxime, adipaldehydic acid oximes, and 6-aminohexanoic acid are given and interpreted.

L21 ANSWER 12 OF 13 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 1906:163714 CAPLUS
DOCUMENT NUMBER: 0:163714
TITLE: On oxidations by means of ozone. [machine translation]
AUTHOR(S): Harries, C.
CORPORATE SOURCE: I. Chem. Inst. d. Univ., Berliu
SOURCE: Berichte der Deutschen Chemischen Gesellschaft (1903), 36, 1933-36
From: Chem. Zentr., 1903, II, 188-189
CODEN: BDCGAS
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

AB [Machine Translation of Descriptors]. In the technology for a long time, ozone in larger scales are used than oxidants, for example, for the preparation of soluble strength and for the production of vanillin from isoeugenols, one of this medium was already rarely availed for scientific purposes. The principal reason for this lies probably in the fact that the SIEMENS and BERTHELOT tubes existing in the laboratories are not sufficient for the preparation of larger quantities of ozone. The author

used the subsequent experiments with the larger, by SIEMENS & HALSKE apparatus which can be referred, in which the oxygen with currents of 5000 to 6000 volts voltage was ozonized. In such a way, gas mixture produced was proved as excellent medium for the preparation of aldehydes from unsaturated compounds. One leads strongly ozonized oxygen into very well cooled Mesityl oxide, (CH3) 2C: CH·CO·CH3, then a thick, stinging smelling, yellow oil forms, which when taken out of the refrigerant, heats mixture with lower rattling noise automatically and then lower fire feature vehemently exploded. Certainly peroxide could be present here, (see original document for formula). Completely smooth and safe, the oxidation of the Mesityl oxide was carried out against it with the presence of water, lower formation of acetone, (CH3)2CO, and methyl glyoxal, CH3·CO·CHO. Analogous produces Methyl heptenone, (CH3)2C : CH·CH2·CH2·CO·CH3, acetone and Laevulin aldehyde (Pentanonal),CH3·CO·CH2·CH2·CHO. By the author, (Ber. German Chemistry Society vo. 31, pg. 37; C. vo. 98, I. pg. 448), in early times by cleavage of the Sylvane (α -Methyl furfuran) from the beech tar obtained Ketonaldehyde by means of ozonized oxygen, also represented completely smoothly from allyl acetone, CH2:CH·CH2·CH2· CO·CH3. The Methylphenyldihydropyridazine, (see original document for formula), available from it by condensation with phenylhydrazine diluted acid, melting, against the earlier indication, at 196 to 197° without decomposition. Analogous of these results would have the citral if the TIEMANN formula (CH3) 2C:CH·CH2·CH2·C(CH3): CH·CHO concerning the position of the double bonds is correct, with the oxidation with ozone Pentanonal to likewise be produced; in reality, however this aldehyde developed only in very small quantity, a very decomposable dialdehyde or Ketoaldehyde was the major product of the reaction. Also unsaturated aldehydes and acetals was easily contested by ozone. 3g Acrolein acetal, CH2:CH·CH(OC2H5)2, was oxidized for 1 1/2 hours to the Glyoxal semiacetal,CHO·CH(OC2H5)2,; stinging odor liquid; Kp760, 80 to 90° under decomposition, (apparent decay in alcohol and glyoxal); with Phenylhydrazinacetate, produces an oily hydrazone, that when heated, turns to the gold-yellow needles of the Glyoxalphenylosazone, solidifies to C6H5·NH·N : CH·CH:N·NH·C6H5. So far, oleic acid, cinnamic acid, maleic acid, and the Fumaric acid dimethyl ester were oxidized by unsaturated acid with ozone. The results with acid are not yet completely clear, the cinnamic acid slowly turned into benzaldehyde and glyoxylic acid, CHO·COOH. the preparation of latter acid could be more recommendable than the easily soluble maleic acid. When Fumaric acid ester suspends 2g, in 20 ccm water, were treated with ozone for about 2 hours, remained 1.4g of esters unmodified, during from the filtrate by Phenylhydrazinacetate 1g Glyoxylic acid methyl ester phenyl hydrazone, CH3OOC·CH:N·NH·C6H5, was precipitated. Gold-yellow needles from diluted methyl alcohol with melting point of 139°. Also alcohols are oxidized by ozone. Methyl alcohol in 50% solution produced formaldehyde; Glycerol in a solution of Phenylhydrazinacetate, produced the known osazone CH2 (OH) ·C(: N·NH·C6H5)·CH : N·NH·C6H5, the glyceraldehyde, CH2(OH)·CH(OH)·CHO. Dioxyacetone, CO(CH2·OH)2, was produced. Stilbene, C6H3·CH : CH·C6H5, was cleaved with the presence of water slowly, but smoothly to benzaldehyde. The oxidation with ozone, which would be allowed to make the easier production for a number was possible so far with difficulty accessible dialdehydes, Ketoaldehyde and Aldehyde acid, it

is to be implemented always with caution, since some compounds let most explosive peroxides develop as intermediates. It is strange that these oxidations remain always standing with the formation of aldehydes: in no case could be isolated appropriate acid also are only in traces.

L21 ANSWER 13 OF 13 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1906:151846 CAPLUS

DOCUMENT NUMBER: 0:151846

TITLE: Over the Effect of Ozone on Organic Compounds. [machine translation]

AUTHOR(S): Harries, C.

CORPORATE SOURCE: Kiel. Chem. Lab. der Univ.

SOURCE: Justus Liebigs Annalen der Chemie (1905), 343, 311-75
From: Chem. Zentr., 1906, I, 542-547
CODEN: JLACBF

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

AB [Machine Translation of Descriptors]. I. Paper. In the introduction discusses the author work of the authors, with the effect of ozone on organic compounds employed. After these ozone of hydrocarbons, like methane and ethyl one, can attack furthermore alcohols under formation of aldehydes, acid and H2O2; sometimes also peroxide-like, explosive compounds of unknown quantity composition could be isolated, which seemed to decompose with water in complicated way. Like the author in the subsequent summary of its experiments implemented with a set of employees (Ber. Dtsch. Chemical Ges. vo. 36, pgs. 1933, 2996, 3658; C. vo. 1903, II. pgs. 188, 932, 1311; Ber. Dtsch. Chemical Ges. vo. 37, pgs. 612, 839, 842, 845, 3431; C. vo. 1904, I. pgs. 924, 1043, 1044, 1045; II. pg. 1112; Ber. Dtsch. Chemical Ges. vo. 38, pg. 1195, 1630; C. vo. 1905, I. pgs. 1244, 1529) show, form except unsaturated hydrocarbons also unsaturated alcohols of ozonides with a molecule O3, on the other hand unsaturated ketones, aldehydes take up and monobasic acid 4 atoms O, by 1 mole O3 to the double bond C : C deposits and fourth O to the carbonyl C : O steps. Latter acceptance finds its confirmation by the preparation of the peroxide of a saturated compound, the oenanthols. For the constitution of the ozonides O the subsequent view unsaturated carbonyl compounds with 4 atoms use. The cleavage such of a compound, for example of the ozonide of the mesityloxide by water will run according to the pattern given in former times in the subsequent way: By introducing O3 in methylheptenone with exclusions from water, as then general with compounds with the grouping becomes R-(CH3)C : C the formation of acetone superoxide, and the analogous compounds observes. It is a breakup takes place thus here to the ozonide group: (see original document for graphics). With this cleavage no H2O2 should arise. With the mesityloxide and methyl heptenonozonide, meanwhile H2O2 is proven; its formation is to be led back to fourth oxygen atoms bound at carbonyl: The description of the characteristics of the ozonides given already in former times still their cleavage is through Br and their reduction with aluminum amalgam in ether solution to acid, and to attach aldehydes or ketones. Generally thus O3 can affect organic bodies in the following two different ways. I. The molecule ozone deposits as O3; ozonides develop (for example with unsaturated carbon compounds). II. The molecule ozone splits during the effect; it forms unstable peroxides (for example accumulation to the CO group). The author uses the facts found by him for the explanation of the reaction process of older investigations, like the oxidation of ethylene by O3, the transfer of the alcohols into aldehydes. After experiments of the author with ethyl peroxide (BERTHELOT,

C. R. D. l'Acad. des sciences vo. 92, pg. 895) neither the composition (C2H5) 4O3, nor is it a peroxide, (C2H5)2O : O, or an ozonide, (C2H5) O·O3. Ozone oxidation and autoxidation are different processes. Very usefully the ozone method has for the preparation of with difficulty accessible preparations, for example of the laevulinaldehyde, as well as for constitution determination, for example of the 1-phenylbutene, proved. Like in former times for the mark in and fumaric acid (HARRIES, Ber. Dtsch. Chemical Ges. 36. 1933; C. 1903. II. 188) is recently from LANGFELD (Dissert. Berlin 1904) the stereoisomerism of the crotone and the isocrotonic acid (WIS-LICENUS, LIEBIG's Ann. vo. 248, pg. 281) proven by the fact that both by O3 into presence of water into glyoxalic acid and acetaldehyde disintegrate to acid. In analogous way also the stereoisomerism of oleic acid and the elaidic acid (see below) was determined. Pulegone supplies an ozonide to chloroform; with the oxidation in presence of water the pulegone gives an aldehyde-like compound, which changes homologous acid at air into methyl adipic acid or instead of the expected methylcyclohexandion and acetone. Pinene takes up ozone to chloroform plentifully; the main part of the ozonide here formed seems to be a product that from cleavage of the bicyclic ring resulted. For the constitution question of the benzene and its derivatives it results from experiments of the author that the formation of a benzene triozonide (I.), (see original document for graphics), for the KEKUL.acte.E; and against the center formula of the benzene. Likewise, that can be judged homolog of the benzene, as well as the diphenyl, which a tetrazonide (II.), (see original document for graphics), forms, the presence aliphatic double bonds from the behavior. Naphthalene gives a diozonide (III.), (see original document for graphics), in which the two molecules O3 are adherent to the same ring. One could from this to assume that compound/connection conditions in the two rings of the naphthalene (see in addition BAMBERGER, LIEBIG's Ann. vo. 257, pg. 1; BAMBERGER, LANGFELD, Ber. Dtsch. Chemical Ges. vo. 23, pg. 1124) different is, and naphthalene the structure IV. attributed, (see original document for graphics). It seems permissible, for the benzene derivatives, ozone add, a compound/connection way after aliphatic structure to assume for the central structure of the core passive against O3. At experiments for the reduction of the danger of explosion in CO2-atmosphere one worked. When solvent served, to the extent necessary, chloroform, from which during longer effect of the O3 small quantities of COCl2 develop. To the purification the ozonides can in few acetic esters dissolved and with petroleum-ether precipitated. Concerning the equipment, in particular the ozone apparatus is referred to the original. Experimental Part. The ozonides were extracted by introducing O3 into the carefully dried substances under strong cooling. The operation requires on the average 2 hours for ever 3 g. Most ozonides are in ether, benzene, and acetic ester and chloroform easily soluble, in petroleum-ether and ligroin little soluble Allyl alcohol ozonide (V), (see original document for graphics), syrup, decomposing at usual temperature; aldehyde splitting suffers when cooking with water; seems to disintegrate with distillation in the vacuum in similar way. Secondary methylheptenolozonide (VI.), (see original document for graphics), oil; D25,5, 1.0992; aldehyde splitting suffers when cooking with water . Tertiary methylhexenol, C7H14O, from allyl acetone and methyl magnesium iodide formed, is a pleasantly odorous liquid of the Kp16, 57°. Ozonide (VII.), (see original document for graphics). Liquid, D23,5, 1.0703; aldehyde splitting suffers when cooking with water. Tertiary dimethylheptenolozonide (VIII.) (see original document for graphics). D23.5, 1.0603; splits when cooking with water into aldehyde and acetone. Allylacetone ozonide (IX.), (see original document

for graphics), very explosive syrup, D19, 1.1814. Disintegrates when cooking with water into HCHO, laevulinaldehyde and H2O2 (see HARRIES, Ber. Dtsch. Chemical Ges. vo. 36, pg. 1933; C. vo. 1903, II. pg. 188). Methylheptenonozonide (X.), (see original document for graphics). The ozonization of the methylheptenone results in one of acetone superoxide interspersed syrup; D19, 1.138. With 10 hours ozonizing of 10 g methylheptenone and cooking that of ozonide with 20 ccm water will form 4 to 5 g laevulinaldehyde beside acetone superoxide. The citronellalozonide, C10H18O5, is a little explosive syrup, D21, 1.0746; disintegrates with distillation in the vacuum partly into the aldehyde and O; with the cleavage with water a ketonaldehyde, C9H16O2 seems to form. The citralozonide, C10H16O5, is a syrup of D21, 1.1486°; it disintegrates when warming up with water. With the two last ozonides the molecular weight determined shows large irregularities of the monomolecular formula. The isocrotonic acid ozonide (XI.), (see original document for graphics), forms yellowish, extremity explosive syrup, for the O-development of isocrotonic acid under when standing regenerated and with water under hissing decomposing. By 1-hour ozonizing of a solution of 2 g of isocrotonic acid in water is split the acid into acetaldehyde and glyoxylic acid: CH3·CH : CH·COOH +O3 + H2 = CH3·CHO + CHO·COOH + H2OO, and hereby their constitution proved. The oenanthylperoxide, CH3·(CH2)5·CH : O : O, by 2-hours ozonizing formed from 2 g oenanthol, is in alcohol, ether, benzene and glacial acetic acid easily soluble oils of ozonide smell, D8, 0.9081; nD8 = 1.4286; decomposing with water in oenanthol and H2O2, forms under self-heating when standing or by effect of NaOH and oenanthylic acid, splits with cold, diluted H2SO4 oxygen. II. Over the Isomerism of the Oil and Elaidic Acid, (With Carl Thieme). The proof for the stereoisomerism of oleic acid and the elaidic acid, CH8·(CH2)7· (CH2)7·COOH, relies so far on the observation that both from dibromo oleic acid and from the dibromelaidine acid, the same Stearolic acid develops. By decomposition of the ozonides both acids by water, which after the equation: (see original document for equation), runs, the authors have the justification of the past acceptance proved. The solution of 5 g oil acid Na in 100 ccm water becomes about 4 to 5 hours ozonized and in the vacuum evaporated. The distillate become by ether the nonylaldehyde, C8H17·CHO (WALBAUM and STEPHAN, Ber. Dtsch. Chemical Ges. vo. 33, pg. 2502; C. vo. 1900, II. pg. 860), and the pelargonic acid, C8H17·COOH, extracted, which can be separated by fractional distillation of under 15 mm pressure. From the backward alkaline solution diluted H2SO4 and ether become the azelaic acid, by acidifying with COOH·(CH2)7·COOH (melting point 106°)form. Semicarbazone of the nonylaldehyde, C9H18 : N·NH·CO·N H2. Glossy lamellas (from methyl alcohol); melting point 84°. Due to the sparingly soluble and the dissociation of the alkaline salts of the elaidic acid with water the above experiment cannot be implemented with these acid. The ozonide of the elaidic acid (see equation), with 4 to 5-hours ozonizing of the solution formed from 5 g of acid in 100 ccm chloroform, is a gelatinose mass, which burns platinum plate down without explosion on that; with heating up with water it gives the H2O2 test, reduces FEHLING solution, oxidizes Ki and colored to indigo and KMnO4-solution. The ozonide of oleic acid resembles the evenly described perfectly. 1 to 2 hours the ozonides are cooked with water which gives an oil, from its ether solution after the shaking with NaHCO3 and water of the nonylaldehyde and the pelargonic acid were isolated. Pelargonic acid, Ca·[CH5·(CH2) 7·COO]2. Powder, melting point

216° - Ag·CH3 (CH2)7·COO. Light sensitive powders.
In the powder, which possesses a weakly rancid smell and which shows aldehyde reactions, extracted by acidifying the $NaHCO_3$ solution, and ether, the half aldehyde of the azelaic acid becomes as semicarbazone, COOH·(CH2)7· CH·N·NH·CO·NH2,
melting point 163°, isolates, and the azelaic acid (melting point 106.5°) proven. III. Over the Determination of the Constitution of some Hydrocarbons by Ozone. The 1,5-hexadiendiozonide, $C_6H_{10}O_6$, becomes with 4 to 5-hours introduce from ozone into the chloroform solution of the diallyl, CH2 : CH·(CH2)2· CH:CH2, and evaporation of the chloroform with 20° form as the more viscous very explosive syrup. Warms up the ozonide with water to the water bath and vaporization of the solution at if possible low temperature and pressure gives one with crystals of succinic acid interspersed syrup, with 55° under 10 mm pressure distilled and the characteristics of the succinaldehyde (HARRIES, Ber. Dtsch. Chemical Ges. vo. 34, pg. 1488; C. vo. 1901, II. pg. 186) shows. The reactions prove the constitution of the diallyls. Of HARRIES and WEIL (Ber. Dtsch. Chemical Ges. vo. 37, pg. 845; C. vo. 1904, I. pg. 1145) represented hydrocarbon C_9H_{16} the constitution of a 2,6-dimethylheptadiens (2,5), (CH3)2C: CH·CH2·CH: C(CH3)2, attributed, since its ozonide with water did not split laevulinaldehyde off. Latter observation proved as incorrect and has its reason in the unequally large ruggedness of the two ozonide groups. The hydrocarbon comes to the subsequent experiments the formula of the 2,6-Dimethylheptadiens (2,6), (CH3)2C : CH· CH2·CH2·C (CH3) : CH2, and its ozonide, $C_9H_{16}O_6$, with 8-hours introduce, formed from O_3 into the chloroform solution from 15 g of the hydrocarbons, a syrup is and gives when cooking with water the laevulinaldehyde, that as phenylmethyldihydropyrazine, $C_{11}H_{12}N_2$ (HARRIES, Ber. Dtsch. Chemical Ges. vo. 31, pg. 45; C. vo. 98, I. pg. 448) one identifies. The hydrocarbon under-laminated with water goes when introducing O8 into solution the same gives the H_2O_2 test and the aldehyde reactions and leaves with the vaporization in the vacuum a very explosive syrup of the presumable composition (XII.), (see original document for graphics). With 1/4-hours succinic acid ethyl esters cook and 500 g absolute from methyl magnesium iodide (24 g Mg, 142 g CH8I) with 44 g. Bernsteinsaureathylester and 500 g absolute alcohol with the 2,5-dimethylhexandiol (2,5), (CH3)2C(OH)· CH2·CH2·C(CH3)2 ·OH, ether formed. Sheets from petroleum-ether melting point 89°; into most organic solvents easily soluble. The glacial acetic acid solution of 60 g diol gives the 2,5-dimethylhexandibromide to glacial acetic acid HBr with 440 g (2,5), (CH3)2CBr·CH2 ·CH2·CBr(CH3) 2; it melting after recrystallizing from little methyl alcohol with 71° and is into the organic solvents easily soluble; weathered at air under HBr discharged. By 3/4-hours cooking the dry dibromide with the 5-fold quantity pyridine an oil one extracts, from which with strong cooling crystals (V) separate, (see original document for formula). The main part of the oil forms with -21° not solidifying 2,5-dimethylhexadiene (1,5), CH2 : C(CH3)·CH2· CH2·C(CH8) : CH2. Easily mobile, unpleasantly smelling liquid, Kp755, 137°. With repeated distillation the boiling point sinks suddenly to 115-117°. From its behavior to O_2 the constitution of the hydrocarbon results; gives with EEC-HBr with 71° melting dibromide. By introducing ozone into the strongly cooled hydrocarbon the ozonide, $C_8H_{14}O_6$, became when more yellowish, more viscous, very explosive syrup formed, the formaldehyde formation decomposing under when standing. During the effect of O_3 on the hydrocarbon laminated over water a solution develops for under formaldehyde formation, in which H_2O_2 and methyl acetone are proven as

diphenylhydrazone. The crystals separated from the crude oil (see above) are an isomer hydrocarbon, C8H14, with about -5° melting and with 142° boiling at air it oxidizes easily to the compound C8H14O2 (?) and a not solidifying oil of the composition gives C8H15Br with glacial acetic acid HBr, which decolorizes Br in glacial acetic acid and KMnO4. IV. Over Ozonides of the Aromatic Hydrocarbons. By effect of O8 on toluene with 0° develops after RENARD (C. R. D. l'Acad. des sciences vo. 121, pg. 651) the ozotoluole, C7H8O6, which are very similar to the RENARD benzene triozonide, and with 8-10° decomposed. During the repetition the authors a compound C7H8O7, which possessed the characteristics of the RENARD ozotoluole, formed experiment, but a decomposition product of the toluoltriozonide was already. This seems with -21° from toluene and ozone to form, durable and exploded at usual temperature is not very vehement. Methylglyoxal and glyoxal seems to contain the solution formed when cooking the RENARD ozotoluole with water. The m-xylolozonide is able to exist only at very low temperature; with 0° prepared substance is a decomposition product. The mesitylenozonide, C6H8(CH8) 3(O3)8, is a gelatinose mass, which explodes still damp with room temperature. After standing lasting several hours of the ozonide with water with 0° and following boiling up could from the solution by semicarbazidchlorohydrate and K-acetate the Methylglyoxyldisemicarbazone (melting point 255 to 257°) are isolated. By introducing O3 into the chloroform solution of naphthalene and washing the glassy residue residual with evaporating the chloroform with ether the naphthalene diozonide (III.) in very explosive crystals was formed, (see original document for graphics). In the solution extracted by decomposition of the ozonide with water became the phtaldialdehyde (Thiele, WINTER, LIEBIG's Ann. vo. 311, pg. 360; C. vo. 1900, II. pg. 376) beside phtalic acid proven. Phenanthrene gave, in which with naphthalene treats indicated way with O8, the very explosive crystals of the phenanthrendiozonide, to C14H10O6. The diphenyltetraozonide (II.), (see original document for graphics), is a colorless, crystallinic mass, which explodes with heating up strongly. Appendix: Over the Ethyl Peroxide of BERTHELOT. By introducing lasting several hours of O3 in strongly cooled, absolute dry ether with presence of CO2, evaporation of the excess ether in the vacuum and distillation of the residue with 20 mm pressure went with 40 to 50° ethyl peroxide as colorless, viscous mass over, which separated very vehement exploded during strong cooling of crystals and occasionally with admission of air. It showed a very low carbon content [15.28 to 25.65%; after BERTHELOT's formula (C2H5)4O3, C = 58.54%]. On that platinum plate deflagrated it, gives when cooking with water acetaldehyde and H2O2, with concentrated KOH ozone smell.

=> FIL STNGUIDE

COST IN U.S. DOLLARS	SINCE FILE ENTRY	TOTAL SESSION
FULL ESTIMATED COST	282.11	282.32

DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)	SINCE FILE ENTRY	TOTAL SESSION
CA SUBSCRIBER PRICE	-36.00	-36.00

FILE 'STNGUIDE' ENTERED AT 17:35:24 ON 16 JAN 2008

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FILE CONTAINS CURRENT INFORMATION.
LAST RELOADED: Jan 11, 2008 (20080111/UP).

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     (FILE 'HOME' ENTERED AT 16:51:32 ON 16 JAN 2008)

     FILE 'CAPLUS' ENTERED AT 16:51:59 ON 16 JAN 2008
L1              9 S ORGANIC (L) OZONIDES (L) (MAKE OR PRODUCE)
L2            203 S OZONIZED (L) (FAT? OR OIL?) (L) WATER
L3              0 S L2 AND (PRODUCE OR MAKE) (L) PEROXICE
L4              3 S L2 AND (PRODUCE OR MAKE) (L) PEROXIDE
L5              0 S (OIL AJD WATER) (L) (OZONE OR OXYGEN) AND PEROXIDE
L6            268 S (OIL (4W) WATER) (S) (OXYGEN OR OZONE)
L7             16 S L6 AND PEROXIDE
L8             16 S L7 NOT L4 NOT L1
L9              7 S L8 AND (PROCESS OR MAKE)
L10           852 S (FAT# OR OIL#) (S) WATER (S) (OZONE OR OXYGEN)
L11             2 S L10 AND (PEROXIDE (5W) PRODUCT)
L12             0 S (SUNFLOWER ADJ OIL)(S) WATER (S) (OZONE OR OXYTEN)
L13            11 S (VEGETABLE (A) OIL) (S) WATER (S) (OZONE OR OXYGEN)
L14             0 S L13 NOT L10
L15            10 S L13 NOT L8
L16             1 S (SUNFLOWER (A) OIL) (S) WATER (S) (OZONE OR OXYGEN)
L17            47 S OZONIZED (S) (FAT# OR OIL) AND PEROXIDE
L18            47 S L17 NOT L13
L19            47 S L17 NOT L7
L20            47 S L19 NOT L15
L21            13 S L20 AND WATER

     FILE 'STNGUIDE' ENTERED AT 17:35:24 ON 16 JAN 2008
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